

## STIC Database Tracking Number

To: GREGORY LISTVOYB  
Location: REM-10D60  
Art Unit: 1796  
Wednesday, December 19, 2007  
Phone: (571) 272-6105  
Case Serial Number: 10 / 560861

From: JAN DELAVAL  
Location: EIC1700  
REM-4B28 / REM-4A30  
Phone: (571) 272-2504

jan.delaval@uspto.gov

## Search Notes

1-7

**Anekwe, Imelda (ASRC)**

046053

**From:** GREGORY LISTVOYB [gregory.listvoyb@uspto.gov]  
**Sent:** Monday, December 17, 2007 12:47 PM  
**To:** STIC-EIC1700  
**Subject:** Database Search Request, Serial Number: 10/560861

**Requester:** GREGORY LISTVOYB (P/1796)  
**Art Unit:** GROUP ART UNIT 1796  
**Employee Number:** 83146  
**Office Location:** REM 10A61  
**Phone Number:** (571)272-6105  
**Mailbox Number:** RA61

**Case serial number:** 10/560861  
**Class / Subclass(es):**  
**Earliest Priority Filing Date:**  
**Format preferred for results:** Paper  
**Attachments:** No attachment.  
**Search Topic Information:**

**Claims 1-7**  
**Special Instructions and Other Comments:**

TO: EIC1700  
FROM: STIC-EIC1700  
SUBJECT: Database Search Request, Serial Number: 10/560861

DEC 17 -

Pat. & T.M. Office

Jan  
Milton

12/17/2007



# STIC Search Results Feedback Form

**EIC17000**

Questions about the scope or the results of the search? Contact the EIC searcher or contact:

Kathleen Fuller, EIC 1700 Team Leader  
5711272-2505 REMSEN 4B28

## Voluntary Results Feedback Form

- I am an examiner in Workgroup:  Example: 1713
- Relevant prior art found, search results used as follows

- ☐ 102 rejection
- ☐ 103 rejection
- ☐ Cited as being of interest.
- ☐ Helped examiner better understand the invention.
- ☐ Helped examiner better understand the state of the art in their technology.

Types of relevant prior art found:

- ☐ Foreign Patent(s)
- ☐ Non-Patent Literature  
(journal articles, conference proceedings, new product announcements etc.)

- Relevant prior art not found:

- ☐ Results verified the lack of relevant prior art (helped determine patentability)
- ☐ Results were not useful in determining patentability or understanding the invention.

Comments:

=> fil reg

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Property values tagged with IC are from the ZIC/VINITI data file provided by InfoChem.

STRUCTURE FILE UPDATES: 18 DEC 2007 HIGHEST RN 958693-84-0

DICTIONARY FILE UPDATES: 18 DEC 2007 HIGHEST RN 958693-84-0

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TSCA INFORMATION NOW CURRENT THROUGH June 29, 2007

Please note that search-term pricing does apply when conducting SmartSELECT searches.

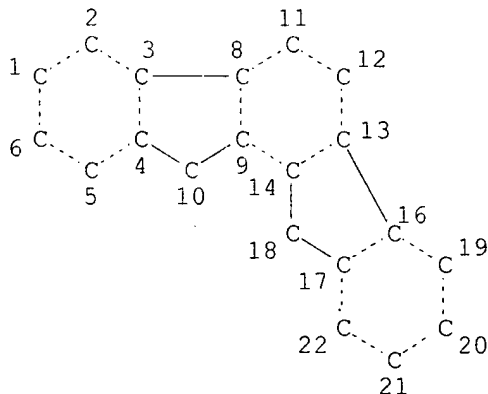
REGISTRY includes numerically searchable data for experimental and predicted properties as well as tags indicating availability of experimental property data in the original document. For information on property searching in REGISTRY, refer to:

<http://www.cas.org/support/stngen/stndoc/properties.html>

=> d sta que l19

L17

STR



NODE ATTRIBUTES:

DEFAULT MLEVEL IS ATOM

DEFAULT ECLEVEL IS LIMITED

GRAPH ATTRIBUTES:

RING(S) ARE ISOLATED OR EMBEDDED

NUMBER OF NODES IS 20

STEREO ATTRIBUTES: NONE

L19 120 SEA FILE=REGISTRY SSS FUL .L17

100.0% PROCESSED 22911 ITERATIONS

120 ANSWERS

SEARCH TIME: 00.00.01

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(FILE 'HOME' ENTERED AT 07:24:36 ON 19 DEC 2007)  
SET COST OFF

FILE 'HCAPLUS' ENTERED AT 07:24:44 ON 19 DEC 2007

L1 1 S US20060149016/PN OR (US2006-560861# OR EP2003-14042)/AP,PRN N  
E ODELL/AU  
E ODELL R/AU  
L2 18 S E3-E5,E8  
E O DELL R/AU  
L3 65 S E3-E9,E12,E13  
E O DELL/AU  
E POUNDS/AU  
E POUNDS T/AU  
L4 16 S E4-E10  
E WALLACE/AU  
L5 4 S E3  
E WALLACE P/AU  
L6 375 S E3-E15,E30-E42  
E TOWNS/AU  
L7 53 S E19-E23  
E MCKIERNAN/AU  
L8 28 S E35-E37,E42-E44  
E MC KIERNAN/AU  
L9 1 S E7  
E COVION/CO  
L10 101 S E4-E6  
L11 83 S E4-E6/PA,CS  
E E4+ALL  
E E1+ALL  
L12 34924 S E2+RN OR E2-E75/PAMCS OR MERCK?/CO,PA,CS  
E COVION/PA,CS  
L13 101 S E3-E20  
SEL RN L1

FILE 'REGISTRY' ENTERED AT 07:29:26 ON 19 DEC 2007

L14 11 S E1-E11  
L15 3 S L14 AND NR>=5  
E 6952/RID  
L16 37 S E6  
L17 STR  
L18 9 S L17  
L19 120 S L17 FUL  
SAV TEMP L19 LISTVOYB560/A  
L20 120 S L15,L16,L19

FILE 'HCAOLD' ENTERED AT 07:32:51 ON 19 DEC 2007

L21 12 S L20

FILE 'HCAPLUS' ENTERED AT 07:33:17 ON 19 DEC 2007

L22 98 S L20  
L23 78 S L22 AND PY<=2003 NOT P/DT  
L24 11 S L22 AND (PD<=20030623 OR PRD<=20030623 OR AD<=20030623) AND P  
L25 89 S L23,L24  
L26 1 S L1-L13 AND L25  
E CONDUCTING POLYMERS/CT  
L27 15526 S E3-E8  
E E3+ALL  
L28 18961 S E5,E6

L29 1 S L25 AND L27,L28  
 E SEMICONDUCTOR DEVICES/CT  
 L30 120106 S E3-E45  
 L31 672291 S E50+OLD,NT OR E56+OLD,NT OR E59+OLD,NT OR E62+OLD,NT OR E73+O  
 E E3+ALL  
 L32 429208 S E7+NT  
 E E6+ALL  
 L33 464207 S E3+OLD,NT OR E79+OLD,NT  
 E SEMICONDUCT/CT  
 L34 1076 S E38+OLD,NT  
 L35 1 S E40  
 E E41+ALL  
 L36 94798 S E2  
 L37 3 S L25 AND L30-L36  
 E SOLAR CELLS/CT  
 L38 21692 S E3-E8  
 E E3+ALL  
 L39 43702 S E13+OLD,NT  
 E PHOTOELECTRIC DEVICES/CT  
 L40 34442 S E3-E30  
 E E3+ALL  
 L41 71142 S E5+OLD,NT  
 E E26+ALL  
 L42 31475 S E10+NT  
 E ELECTROLUMINESCE/CT  
 L43 13417 S E4-E6 OR E4+OLD,NT  
 L44 62416 S E10-E21 OR E10+OLD,NT  
 E E4+ALL  
 L45 13417 S E8+OLD  
 L46 278188 S E7+OLD,NT OR E15+OLD,NT OR E17+OLD,NT  
 E E16+ALL  
 L47 62416 S E18+OLD  
 L48 8 S L25 AND L38-L47  
 L49 4 S L25 AND ?LUMINESC?  
 L50 4 S L25 AND H01L/IPC,IC,ICM,ICS  
 L51 2 S L25 AND C09K011/IPC,IC,ICM,ICS  
 L52 3 S L25 AND H05B033/IPC,IC,ICM,ICS  
 L53 2 S L25 AND C08G061-02/IPC,IC,ICM,ICS  
 L54 1 S L25 AND C08F/IPC,IC,ICM,ICS  
 L55 9 S L26,L29,L37,L48-L54  
 E PHOSPHOR/CT  
 L56 0 S L25 AND E3  
 L57 2 S L25 AND (E33+OLD,NT OR E36+OLD,NT OR E43+OLD,NT)  
 E PHOSPHORS/CT  
 L58 1 S L25 AND E3-E15  
 E E3+ALL  
 L59 1 S L25 AND E4+NT  
 L60 9 S L55-L59  
 L61 16 S L25 AND ?POLYM?  
 L62 7 S L25 AND (POLYM? OR PLASTIC?)/SC,SX,CW,CT  
 L63 4 S L25 AND ?PLASTIC?  
 L64 24 S L60-L63  
 L65 2 S L24 NOT L64  
 L66 26 S L64,L65  
 SEL HIT RN

FILE 'REGISTRY' ENTERED AT 07:54:23 ON 19 DEC 2007

L67 58 S E1-E58  
 L68 22 S L67 AND (C20H1002 OR C32H20BR2 OR C20H10N4 OR C20H12O OR C24H  
 L69 62 S L20 NOT L67

L70 14 S L69 AND L15,L16  
L71 48 S L69 NOT L70  
L72 1 S L71 AND C22H14  
L73 37 S L68,L70,L72

FILE 'HCAPLUS' ENTERED AT 08:03:04 ON 19 DEC 2007

L74 22 S L73  
L75 19 S L74 AND PY<=2003 NOT P/DT  
L76 1 S L73 AND (PD<=20030623 OR PRD<=20030623 OR AD<=20030623) AND P  
L77 20 S L75,L76  
L78 5 S L77 AND ?POLYM?  
L79 3 S L77 AND (POLYM? OR PLASTIC?)/SC,SX,CW,CT  
L80 0 S L77 AND ?PLASTIC?  
L81 1 S L77 AND ?LUMINESC?  
L82 1 S L77 AND (SEMICONDUCT? OR SEMI CONDUCT?)  
L83 1 S L77 AND (C08F OR H01L OR C09K011 OR H05B033 OR C08G061)/IPC,I  
L84 3 S L77 AND L27,L28,L30-L36,L38-L47  
L85 1 S L77 AND L1-L13  
L86 7 S L78-L85,L76  
E ELECTRIC CONDUCTIVITY/CT  
L87 150461 S E3-E32  
L88 80890 S E33-E66  
E E3+ALL  
L89 227304 S E6+OLD,NT  
L90 355779 S E54+OLD,NT OR E55+OLD,NT OR E56+OLD,NT OR E57+OLD,NT  
L91 1 S L77 AND L87-L90  
L92 7 S L86,L91  
L93 3 S L77 AND ELECTR?/SC,SX  
L94 7 S L92,L93  
L95 13 S L77 NOT L94  
L96 20 S L94,L95

FILE 'REGISTRY' ENTERED AT 08:10:04 ON 19 DEC 2007

=> fil hcaplus

FILE 'HCAPLUS' ENTERED AT 08:10:15 ON 19 DEC 2007

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FILE COVERS 1907 - 19 Dec 2007 VOL 147 ISS 26

FILE LAST UPDATED: 18 Dec 2007 (20071218/ED)

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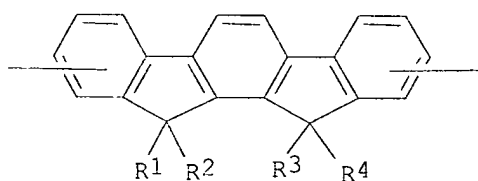
This file contains CAS Registry Numbers for easy and accurate substance identification.

=> d 196 bib abs hitind hitstr retable tot

L96 ANSWER 1 OF 20 HCAPLUS COPYRIGHT 2007 ACS on STN  
 AN 2004:1154360 HCAPLUS  
 DN 142:75323  
 TI Production of **semiconductive** oligomers and **polymers**  
 for use in thin film electronic and optical devices  
 PA **Covion Organic Semiconductors GmbH, Germany**  
 SO Eur. Pat. Appl., 26 pp.  
 CODEN: EPXXDW  
 DT **Patent**  
 LA English  
 FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	EP 1491568	A1	20041229	EP 2003-14042	20030623 <--
	R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, HU, SK				
	WO 2004113412	A2	20041229	WO 2004-EP6721	20040622 <--
	WO 2004113412	A3	20050421		
	W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW				
	RW: BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG				
	EP 1639027	A2	20060329	EP 2004-740151	20040622 <--
	R: DE, FR, GB, NL				
	CN 1768090	A	20060503	CN 2004-80008666	20040622 <--
	US 2006149016	A1	20060706	US 2005-560861	20051215 <--
PRAI	EP 2003-14042	A	20030623	<--	
	WO 2004-EP6721	W	20040622		

GI



AB The present invention relates to new **semiconductive** oligomers and **polymers** (I), a process for their manufacture and their use in thin film electronic and optical devices, such as organic light emitting diodes (OLED) and photovoltaic devices, eg. solar cells and photodetectors; wherein R1, R2, R3, R4, which may be the same or different, are independently selected from hydrogen or a substituent and two or more of R1, R2, R3, and R4 may be linked to form a ring.

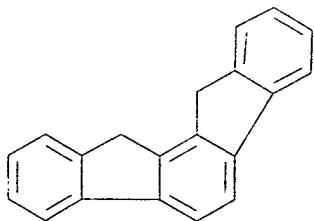
IC ICM C08G0061-02  
 ICS C07C0025-22; H01L0051-30; C07C0001-20; C07C0001-24

CC 37-3 (Plastics Manufacture and Processing)  
 Section cross-reference(s): 52, 73

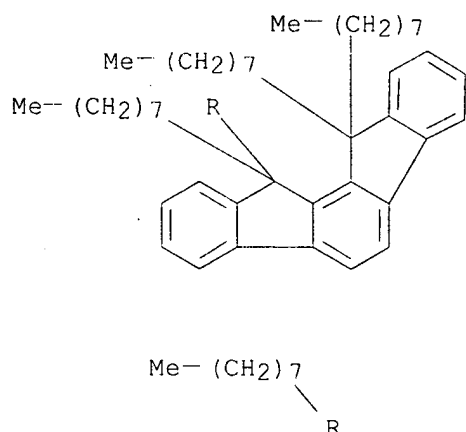
ST **semiconductive** oligomer **polymer** thin film electronic



- optical device
- IT **Conducting polymers**  
(polythiophenes; production of **semiconductive** oligomers and **polymers** for use in thin film electronic and optical devices)
- IT **Conducting polymers**  
**Electroluminescent devices**  
**Optical detectors**  
**Photoelectric devices**  
**Semiconductor devices**  
**Solar cells**  
(production of **semiconductive** oligomers and **polymers** for use in thin film electronic and optical devices)
- IT 1169-58-0P **5815-59-8P** 27609-80-9P, [1,1':4',1''-Terphenyl]-2',3'-dimethanol 49538-77-4P **815588-68-2P**  
RL: IMF (Industrial manufacture); RCT (Reactant); PREP (Preparation); RACT (Reactant or reagent)  
(intermediate; production of **semiconductive** oligomers and **polymers** for use in thin film electronic and optical devices)
- IT **815588-69-3P**  
RL: IMF (Industrial manufacture); RCT (Reactant); PREP (Preparation); RACT (Reactant or reagent)  
(monomer; production of **semiconductive** oligomers and **polymers** for use in thin film electronic and optical devices)
- IT 126213-51-2, Polyethylene dioxythiophene  
RL: TEM (Technical or engineered material use); USES (Uses)  
(production of **semiconductive** oligomers and **polymers** for use in thin film electronic and optical devices)
- IT 111-83-1, Octyl bromide 762-42-5, Dimethylacetylenedicarboxylate 886-65-7, 1,4-Diphenyl-1,3-butadiene 7726-95-6, Bromine, reactions  
RL: RCT (Reactant); RACT (Reactant or reagent)  
(starting material; production of **semiconductive** oligomers and **polymers** for use in thin film electronic and optical devices)
- IT **5815-59-8P 815588-68-2P**  
RL: IMF (Industrial manufacture); RCT (Reactant); PREP (Preparation); RACT (Reactant or reagent)  
(intermediate; production of **semiconductive** oligomers and **polymers** for use in thin film electronic and optical devices)
- RN 5815-59-8 HCAPLUS
- CN Indeno[2,1-a]fluorene, 11,12-dihydro- (CA INDEX NAME)



- RN 815588-68-2 HCAPLUS
- CN Indeno[2,1-a]fluorene, 11,12-dihydro-11,11,12,12-tetraoctyl- (9CI) (CA INDEX NAME)



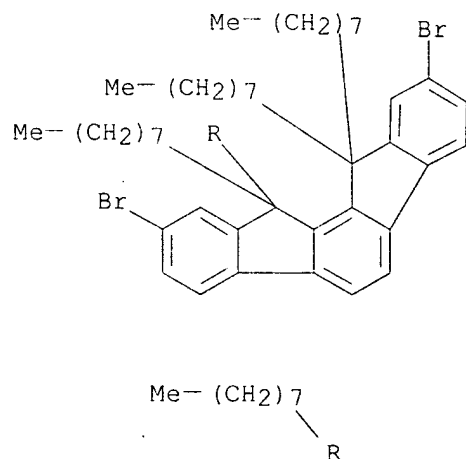
IT 815588-69-3P

RL: IMF (Industrial manufacture); RCT (Reactant); PREP (Preparation); RACT (Reactant or reagent)

(monomer; production of **semiconductive** oligomers and **polymers** for use in thin film electronic and optical devices)

RN 815588-69-3 HCAPLUS

CN Indeno[2,1-a]fluorene, 2,9-dibromo-11,12-dihydro-11,11,12,12-tetraoctyl-(9CI) (CA INDEX NAME)



## RETABLE

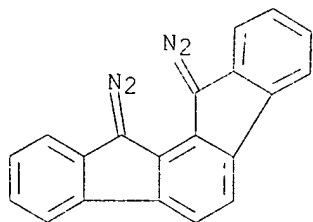
Referenced Author (RAU)	Year (RPY)	VOL (RVL)	PG (RPG)	Referenced Work (RWK)	Referenced File
Harvey, R	2000	165	3952	JOURNAL OF ORGANIC C	
Kreuder, W	2000	1		WO 0022026 A	HCAPLUS
Mullen, K	2000	133	2016	MACROMOLECULES	
Plummer, B	1993	115	11542	JOURNAL OF THE AMERI	HCAPLUS
Robert, T	2000	1		WO 0053656 A	HCAPLUS
Stepanovich, D	1976	1		CH 582115 A	

L96 ANSWER 2 OF 20 HCAPLUS COPYRIGHT 2007 ACS on STN

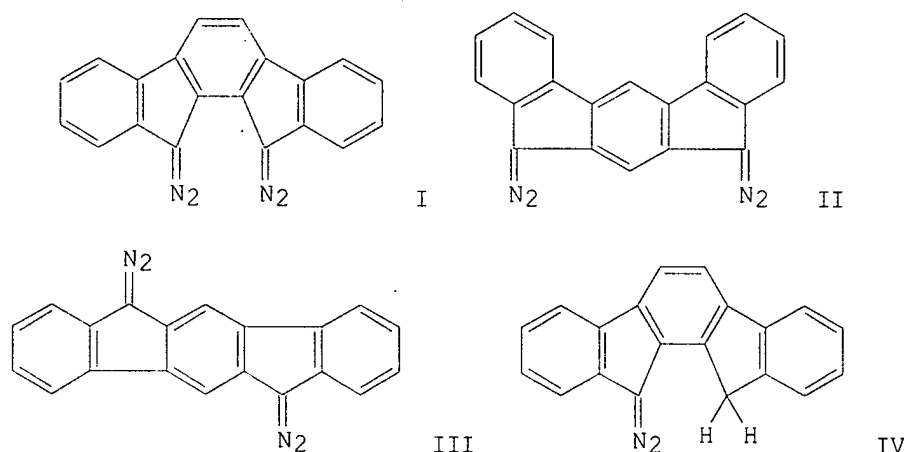
AN 1991:419108 HCAPLUS

DN 115:19108

TI Structure of 11,12-bis(diazo)-11,12-dihydroindeno[2,1-a]fluorene  
AU Miyazaki, Akira; Izuoka, Akira; Sugawara, Tadashi; Bethell, Donald;  
Gallagher, Peter  
CS Coll. Arts Sci., Univ. Tokyo, Tokyo, 153, Japan  
SO Acta Crystallographica, Section C: Crystal Structure Communications (1991), C47(5), 1054-6  
CODEN: ACSCEE; ISSN: 0108-2701  
DT Journal  
LA English  
AB The title compound is orthorhombic, space group Pbcn, with a 14.53(2), b 10.007(9), and c 20.033(4) Å; d.(calculated) = 1.397 for Z = 8. Final R = 0.059 for 1259 reflections. Atomic coordinates are given. The indenofluorene ring is planar and considerable distortion of bond angles and bond lengths is observed in the 2 closely located diazo groups. In the crystal structure, pairs of mols. with antiparallel orientations form a herringbone packing pattern, commonly observed in polycondensed aromatic hydrocarbons.  
CC 75-8 (Crystallography and Liquid Crystals)  
Section cross-reference(s): 25  
IT 92265-85-5  
RL: PRP (Properties)  
(crystal structure of)  
IT 92265-85-5  
RL: PRP (Properties)  
(crystal structure of)  
RN 92265-85-5 HCAPLUS  
CN Indeno[2,1-a]fluorene, 11,12-bis(diazo)-11,12-dihydro- (9CI) (CA INDEX NAME)



L96 ANSWER 3 OF 20 HCAPLUS COPYRIGHT 2007 ACS on STN  
AN 1990:97954 HCAPLUS  
DN 112:97954  
TI Cathodic oligomerization of bis(diazo) compounds of the indenofluorene series. Part 2. Kinetics and mechanistic aspects  
AU Bethell, Donald; Gallagher, Peter; Self, David P.; Parker, Vernon D.  
CS Dep. Chem., Univ. Liverpool, Liverpool, L69 3BX, UK  
SO Journal of the Chemical Society, Perkin Transactions 2: Physical Organic Chemistry (1972-1999) (1989), (8), 1105-9  
CODEN: JCPKBH; ISSN: 0300-9580  
DT Journal  
LA English  
OS CASREACT 112:97954  
GI



AB The time dependence of the disappearance of diazo groups in bis(diazo)indeno[1,2-b]fluorenes I and II during constant-current electrolysis in DMF at a Pt cathode, giving oligomeric polyazines, was examined. The form of the diazo-group concentration-vs.-time curves shows that chain reactions are involved, and the slopes place limits on the chain lengths. Interrupting the current after partial reaction of I showed, by an adaptation of methodol. previously developed to explain the electrochem. reduction of 9-diazo[1,2-b]fluorene, that the chain-termination process is 1st order in the chain carrier; kinetic parameters for the propagation and termination processes in homogeneous solution were obtained. The discrepancy between the observed concentration/time curve for continuous electrolysis and that calculated from

these kinetics parameters gives information on the initiation step, indicating a chain carrier formed from 4 monomer units. Similarly, the results for II best fit a reaction scheme involving, in the early stages, a trimeric chain carrier which is partitioned between chain transfer to monomer and termination by dimerization. Details of these processes are discussed, and the way in which the mechanistic schemes permit interpretation of the differing mol.-weight distributions in the final products from I and II are explained. Supporting evidence is provided by thermolytic studies of I-III in solution and voltammetric expts. on the related monodiazo compound IV.

CC 22-13 (Physical Organic Chemistry)

Section cross-reference(s): 35, 72

IT Kinetics of **polymerization**

(oligomerization, electrochem., of bis(diazo)indeno[1,2-b]fluorenes)

IT **Polymerization**

(oligomerization, electrochem., of bis(diazo)indeno[1,2-b]fluorenes, mechanism of)

IT 92265-85-5

RL: RCT (Reactant); RACT (Reactant or reagent)

(electrochem. oligomerization of, kinetics and mechanism of, and thermal decomposition of, kinetics of, and photolysis of)

IT 123728-99-4

RL: RCT (Reactant); RACT (Reactant or reagent)

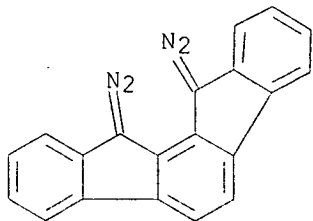
(electrochem. reduction of, kinetics and mechanism of)

IT 92265-85-5

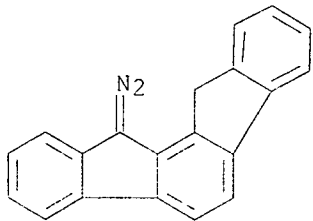
RL: RCT (Reactant); RACT (Reactant or reagent)

(electrochem. oligomerization of, kinetics and mechanism of, and thermal decomposition of, kinetics of, and photolysis of)

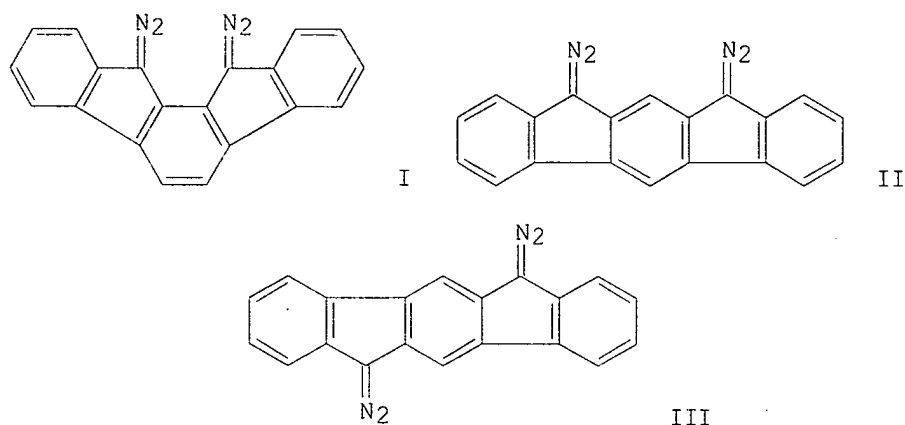
RN 92265-85-5 HCAPLUS  
CN Indeno[2,1-a]fluorene, 11,12-bis(diazo)-11,12-dihydro- (9CI) (CA INDEX NAME)



IT 123728-99-4  
RL: RCT (Reactant); RACT (Reactant or reagent)  
(electrochem. reduction of, kinetics and mechanism of)  
RN 123728-99-4 HCAPLUS  
CN Indeno[2,1-a]fluorene, 11-diazo-11,12-dihydro- (9CI) (CA INDEX NAME)



L96 ANSWER 4 OF 20 HCAPLUS COPYRIGHT 2007 ACS on STN  
AN 1989:615018 HCAPLUS  
DN 111:215018  
TI Cathodic oligomerization of bis(diazo) compounds of the indenofluorene series. Part I. General features  
AU Bethell, Donald; Gallagher, Peter; Bott, David C.  
CS Robert Robinson Lab., Univ. Liverpool, Liverpool, L69 3BX, UK  
SO Journal of the Chemical Society, Perkin Transactions 2: Physical Organic Chemistry (1972-1999) (1989), (8), 1097-104  
CODEN: JCPKBH; ISSN: 0300-9580  
DT Journal  
LA English  
GI



- AB Three bis(diazo) compds. of the indenofluorene series (I, II, and III) were prepared and decomposed by an electrochem. initiated chain process in DMF solution. The black solns. so obtained show single-line ESR spectra and electronic absorption spectra extending into the near IR region of the spectrum. In situ electrochem. ESR spectra of electrolyzed solns. of I and II in CH<sub>3</sub>CN are similar to spectra reported for lightly doped conjugated **polymers**. Chemical evidence is presented to show that the products are polyazines and the results of elemental and gel permeation chromatog. anal. suggest ds.p. in the range 2-5 for I and .apprx.17 for II and III; although in the latter instances there is evidence for material of much greater mol. weight. The black solid materials isolated from the electrolysis solns. and containing supporting electrolyte (Me<sub>4</sub>NBF<sub>4</sub>) show specific conductivities of ca. 10<sup>-10</sup> (1b), 10<sup>-9</sup> (2b), and 10<sup>-6</sup> (3b) ohm<sup>-1</sup> cm<sup>-1</sup>, all increasing to 10<sup>-4</sup>-10<sup>-3</sup> on exposure to gaseous SO<sub>3</sub>.
- CC 35-5 (Chemistry of Synthetic High **Polymers**)  
Section cross-reference(s): 36, 72
- IT **Electric conductivity and conduction**  
(of indenofluorene bis(diazo) oligomers, sulfur trioxide doping effect on)
- IT **Polymerization**  
(oligomerization, electrochem., of indenofluorene bis(diazo) compds.)
- IT **Polymers, preparation**  
(polyketazines, oligomers, preparation of, in cathodic oligomerization of bis(diazo) compds. of indenofluorenes)
- IT **123661-41-6P 123661-43-8P 123661-44-9P**  
RL: SPN (Synthetic preparation); PREP (Preparation)  
(oligomeric, preparation and characterization of)
- IT **92265-85-5P 102150-04-9P 123661-42-7P**  
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
(preparation and cathodic oligomerization of)
- IT **123728-94-9P 123728-95-0P 123728-96-1P**  
**123728-97-2P 123728-98-3P 123728-99-4P**  
RL: SPN (Synthetic preparation); PREP (Preparation)  
(preparation of)
- IT **3074-14-4, Indeno[2,1-a]fluorene-11,12-dione 5695-13-6,**  
**Indeno[1,2-b]fluorene-6,12-dione 112865-17-5, Indeno[2,1-b]fluorene-**  
**10,12-dione**  
RL: RCT (Reactant); RACT (Reactant or reagent)  
(reaction of, with hydrazine hydrate)

IT **123661-41-6P**RL: SPN (Synthetic preparation); PREP (Preparation)  
(oligomeric, preparation and characterization of)

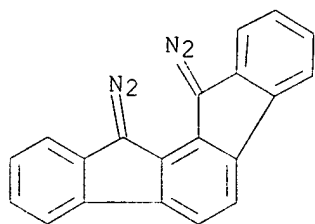
RN 123661-41-6 HCAPLUS

CN Indeno[2,1-a]fluorene, 11,12-bis(diazo)-11,12-dihydro-, homopolymer (9CI)  
(CA INDEX NAME)

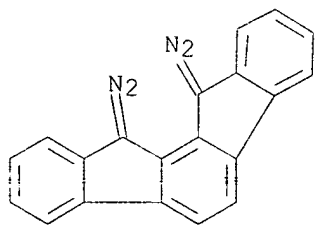
CM 1

CRN 92265-85-5

CMF C20 H10 N4

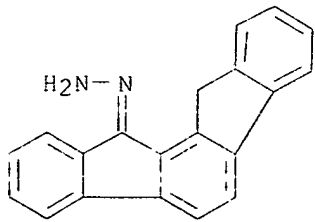
IT **92265-85-5P**RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT  
(Reactant or reagent)  
(preparation and cathodic oligomerization of)

RN 92265-85-5 HCAPLUS

CN Indeno[2,1-a]fluorene, 11,12-bis(diazo)-11,12-dihydro- (9CI) (CA INDEX  
NAME)IT **123728-94-9P 123728-95-0P 123728-96-1P****123728-97-2P 123728-99-4P**RL: SPN (Synthetic preparation); PREP (Preparation)  
(preparation of)

RN 123728-94-9 HCAPLUS

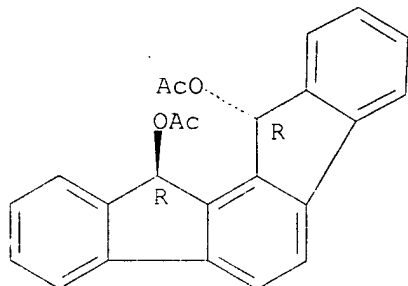
CN Indeno[2,1-a]fluoren-11(12H)-one, hydrazone (CA INDEX NAME)



RN 123728-95-0 HCAPLUS

CN Indeno[2,1-a]fluorene-11,12-diol, 11,12-dihydro-, diacetate, trans- (9CI)  
(CA INDEX NAME)

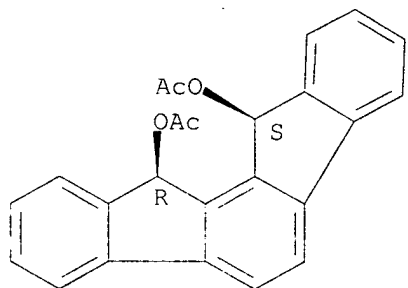
Relative stereochemistry.



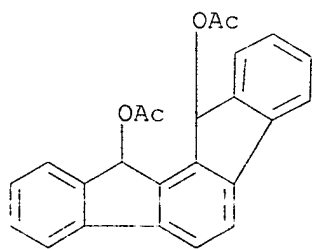
RN 123728-96-1 HCAPLUS

CN Indeno[2,1-a]fluorene-11,12-diol, 11,12-dihydro-, diacetate, cis- (9CI)  
(CA INDEX NAME)

Relative stereochemistry.



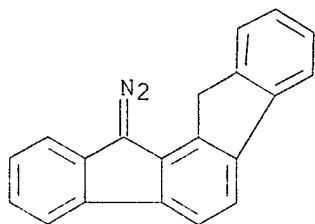
RN 123728-97-2 HCAPLUS

CN Indeno[2,1-b]fluorene-10,12-diol, 10,12-dihydro-, diacetate (9CI) (CA  
INDEX NAME)

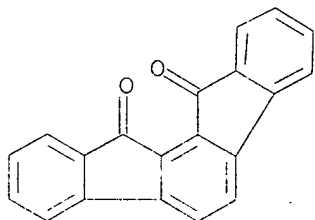
RN 123728-99-4 HCAPLUS

CN Indeno[2,1-a]fluorene, 11-diazo-11,12-dihydro- (9CI) (CA INDEX NAME)





IT 3074-14-4, Indeno[2,1-a]fluorene-11,12-dione  
 RL: RCT (Reactant); RACT (Reactant or reagent)  
 (reaction of, with hydrazine hydrate)  
 RN 3074-14-4 HCAPLUS  
 CN Indeno[2,1-a]fluorene-11,12-dione (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)



L96 ANSWER 5 OF 20 HCAPLUS COPYRIGHT 2007 ACS on STN  
 AN 1989:94360 HCAPLUS  
 Correction of: 1988:509706  
 DN 110:94360  
 Correction of: 109:109706  
 TI Triplet-triplet absorption spectra of organic molecules in condensed phases.  
 AU Carmichael, Ian; Hug, Gordon L.  
 CS Radiat. Chem. Data Cent., Univ. Notre Dame, Notre Dame, IN, 46556, USA  
 SO Journal of Physical and Chemical Reference Data (1986), 15(1), 1-250  
 CODEN: JPCRBV; ISSN: 0047-2689  
 DT Journal  
 LA English  
 AB A review in which a compilation is given of spectral parameters associated with triplet-triplet absorption of organic mols. in condensed media. The wavelengths of maximum absorbance and the corresponding extinction coeffs., where known, were critically evaluated. Other data, for example, lifetimes, energies, and energy transfer rates, relevant to the triplet states of these mols., are included by way of comments, but have not been subjected to a similar scrutiny. An introduction is given to triplet state processes in solution and solids, developing the conceptual background and offering a historical perspective on the detection and measurement of triplet state absorption. Techniques employed to populate the triplet state are reviewed and the various approaches to the estimation of the extinction coefficient of triplet-triplet absorption are discussed. A statistical anal. of the available data is presented and recommendations for a hierarchical choice of extinction coeffs. are made. Data collection is expected to be complete through the end of 1984.  
 CC 22-9 (Physical Organic Chemistry)  
 IT 19840-99-4 20073-24-9 20139-92-8 20266-41-5 20266-45-9

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22362-94-3	22422-70-4	22559-70-2	22559-71-3	22559-72-4
22568-06-5	22970-73-6	22970-75-8	23284-44-8	23489-88-5
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26034-88-8	26071-73-8	26071-74-9	26483-78-3	26754-93-8
26929-86-2	27224-10-8	27876-94-4, 8,8'-Diapo- $\psi,\psi$ -carotenedioic acid	28358-65-8	28406-56-6
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34777-33-8, 1H-Benzo[c]carbazole	34880-57-4	35066-65-0	35139-10-7	35391-47-0
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RL: PRP (Properties)

(triplet-triplet absorption spectrum of)

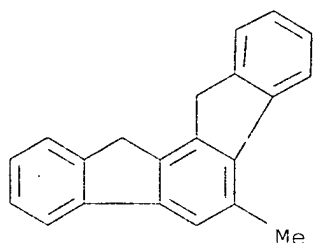
IT **38023-62-0**

RL: PRP (Properties)

(triplet-triplet absorption spectrum of)

RN **38023-62-0** HCAPLUS

CN Indeno[2,1-a]fluorene, 11,12-dihydro-5-methyl- (CA INDEX NAME)



L96 ANSWER 6 OF 20 HCAPLUS COPYRIGHT 2007 ACS on STN

AN 1988:509706 HCAPLUS

Correction of: 1986:552243

DN 109:109706

Correction of: 105:152243

TI Triplet-triplet absorption spectra of organic molecules in condensed phases

AU Carmichael, Ian; Hug, Gordon L.

CS Radiat. Chem. Data Cent., Univ. Notre Dame, Notre Dame, IN, 46556, USA

SO Journal of Physical and Chemical Reference Data (1986), 15(1), 1-250

CODEN: JPCRBV; ISSN: 0047-2689

DT Journal; General Review

LA English

AB A review in which a compilation is given of spectral parameters associated with triplet-triplet absorption of organic mols. in condensed media. Other data, for example, lifetimes, energies and energy transfer rates, relevant to the triplet states of these mols., are included by way of comments, but have not been subjected to a similar scrutiny. An introduction is given to triplet state processes in solution and solids, developing the conceptual background and offering an historical perspective on the detection and measurement of triplet state absorption. Techniques employed to populate the triplet state are reviewed and the various approaches to the estimation of the extinction coefficient of triplet-triplet absorption are discussed. A statistical anal. of the available data is presented and recommendations for a hierarchical choice of extinction coeffs. are made. Data collection is expected to be complete through the end of 1984.

CC 22-9 (Physical Organic Chemistry)

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	31083-73-5	31589-99-8	31700-39-7	31702-08-6	31702-14-4
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66671-26-9	66933-94-6	66933-95-7	66933-96-8	67024-10-6
67168-90-5	67526-83-4	Benzophenazine		67567-20-8 67588-46-9
67605-76-9	67777-65-5	68243-21-0		

RL: PRP (Properties)

(triplet-triplet absorption spectrum of)

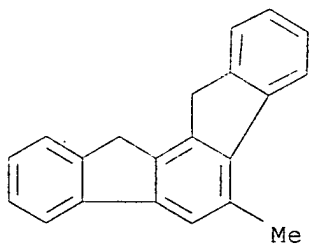
IT **38023-62-0**

RL: PRP (Properties)

(triplet-triplet absorption spectrum of)

RN 38023-62-0 HCAPLUS

CN Indeno[2,1-a]fluorene, 11,12-dihydro-5-methyl- (CA INDEX NAME)



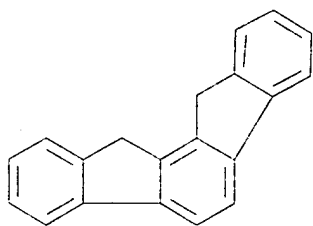
L96 ANSWER 7 OF 20 HCAPLUS COPYRIGHT 2007 ACS on STN

AN 1986:210122 HCAPLUS

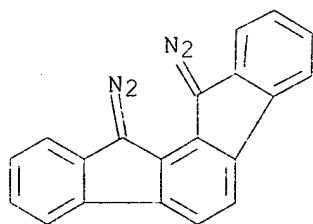
DN 104:210122

TI Characterization of polycyclic aromatic hydrocarbon minerals curtisite, idrialite and pendletonite using high-performance liquid chromatography, gas chromatography, mass spectrometry and nuclear magnetic resonance spectroscopy

AU Wise, Stephen A.; Campbell, Robert M.; West, W. Raymond; Lee, Milton L.;  
Bartle, Keith D.  
CS Cent. Anal. Chem., Natl. Bur. Stand., Gaithersburg, MD, 20899, USA  
SO Chemical Geology (1986), 54(3-4), 339-57  
CODEN: CHGEAD; ISSN: 0009-2541  
DT Journal  
LA English  
AB Two polycyclic aromatic hydrocarbon (PAH) minerals, curtisite [12416-83-0]  
and idrialite [12416-83-0], were characterized using high-resolution gas  
chromatog.-mass spectrometry (GC-MS) and HPLC with fluorescence detection.  
Normal-phase HPLC on an aminosilane column was used to sep. the mineral  
exts. into 6 fractions based on the number of aromatic C atoms in the PAH.  
These fractions were then analyzed by using GC-MS and reversed-phase HPLC  
with fluorescence detection to sep. and identify the individual  
components. One fraction was also analyzed by using NMR spectroscopy to  
provide structural information and information on the position of alkyl  
substitution. Using these anal. techniques, the curtisite and idrialite  
were found to be unique complex PAH mixts. consisting of 6 sp. PAH  
structural series with each member of a series differing from the previous  
member by addition of another aromatic ring. The curtisite and idrialite  
samples contained many of the same components but in considerably  
different relative amts. The major PAH constituents of the curtisite  
sample were: picene [213-46-7], dibenzo[a,h]fluorene [239-85-0],  
11H-indeno[2,1-a]phenanthrene [220-97-3], benzo[b]phenanthro[2,1-  
d]thiophene [55969-62-5], indenofluorenes, chrysene [218-01-9], and  
their methyl- and dimethyl-substituted homologs; the major components in  
the idrialite sample were higher-mol.-weight PAH, i.e. benzonaphthofluorenes  
(mol. weight 316), benzoindenofluorenes (mol. weight 304) and benzopicene  
[220-77-9] (mol. weight 328), in addition to the compds. found in the curtisite  
sample. The combination of HPLC to isolate sp. PAH groups and the anal.  
of these fractions by HPLC-fluorescence and GC-MS resulted in the pos.  
identification of .apprx.20 of the >100 PAH found in these 2 samples. The  
identification of these compds. supports the conclusions of M. Blumer  
(1975) that these minerals were formed by medium-temperature pyrolysis of  
organic compds., followed by extended equilibration at elevated temps. in the  
subsurface.  
CC 53-1 (Mineralogical and Geological Chemistry)  
IT 53-70-3 86-74-8 213-46-7 218-01-9 220-77-9 220-97-3 221-11-4  
238-84-6 239-01-0 239-35-0 239-85-0 239-88-3 239-94-1 243-17-4  
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102124-14-1 102124-15-2 102124-16-3 102124-17-4 102124-18-5  
102124-19-6 102124-20-9 102124-21-0 102124-22-1 102124-23-2  
102137-62-2  
RL: OCCU (Occurrence)  
(in curtisite and idrialite, gas chromatog. and HPLC identification of)  
IT **5815-59-8**  
RL: OCCU (Occurrence)  
(in curtisite and idrialite, gas chromatog. and HPLC identification of)  
RN 5815-59-8 HCAPLUS  
CN Indeno[2,1-a]fluorene, 11,12-dihydro- (CA INDEX NAME)

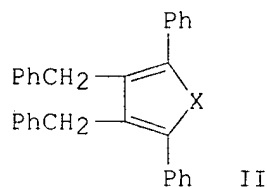


L96 ANSWER 8 OF 20 HCAPLUS COPYRIGHT 2007 ACS on STN  
AN 1984:551229 HCAPLUS  
DN 101:151229  
TI Photolysis of 1,12-bis(diazo)-1,12-dihydroindeno[2,3-a]fluorene. ESR and optical detection of a  $\sigma$ -type 1,4-biradical  
AU Sugawara, Tadashi; Bethell, Donald; Iwamura, Hiizu  
CS Dep. Appl. Mol. Sci., Inst. Mol. Sci., Myodaiji, 444, Japan  
SO Tetrahedron Letters (1984), 25(22), 2375-8  
CODEN: TELEAY; ISSN: 0040-4039  
DT Journal  
LA English  
AB Photolysis of the title bis(diazo) compound in 2-methyltetrahydrofuran glasses at cryogenic temperature has been shown by ESR and UV studies to result in a stepwise cleavage of two nitrogen mols. to give a 1,4- $\sigma$ -biradical.  
CC 22-8 (Physical Organic Chemistry)  
IT 92265-85-5  
RL: RCT (Reactant); RACT (Reactant or reagent)  
(photolysis of)  
IT 92265-85-5  
RL: RCT (Reactant); RACT (Reactant or reagent)  
(photolysis of)  
RN 92265-85-5 HCAPLUS  
CN Indeno[2,1-a]fluorene, 11,12-bis(diazo)-11,12-dihydro- (9CI) (CA INDEX NAME)



L96 ANSWER 9 OF 20 HCAPLUS COPYRIGHT 2007 ACS on STN  
AN 1979:38738 HCAPLUS  
DN 90:38738  
OREF 90:6235a,6238a  
TI Carbocyclizations and heterocyclizations of ortho-dibenzyl derivatives by the effect of sulfur  
AU Lepage, Lucette; Lepage, Yves  
CS Lab. Chim. Org. A, UER Sci. Exactes Nat., Limoges, Fr.  
SO Journal of Heterocyclic Chemistry (1978), 15(7), 1185-91  
CODEN: JHTCAD; ISSN: 0022-152X

DT Journal  
 LA French  
 OS CASREACT 90:38738  
 GI



AB 1,4-Diphenyl-2,3-dibenzyl-1,3-butadiene (I) was prepared by treating di-Et oxalate with  $\text{PhCH}_2\text{MgCl}$  and dehydrating  $(\text{PhCH}_2)_2\text{C}(\text{OH})\text{C}(\text{OH})(\text{CH}_2\text{Ph})_2$ . Treatment of I with N-bromosuccinimide gave 5,11-diphenylnaphthacene. I reacted with S to give the thiophene II ( $\text{X} = \text{S}$ ). II ( $\text{X} = \text{Se}$ ) was obtained from I and  $\text{SeO}_2$ . Benzothiophenes and naphthothiophenes were similarly obtained from other o-dibenzyl compds. and S, with benzo[a]aceanthrylene and indeno[2,1-a]fluorene compds. and by-product.

CC 27-8 (Heterocyclic Compounds (One Hetero Atom))  
 Section cross-reference(s): 25, 26

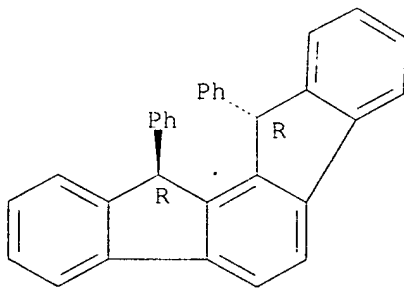
IT 6232-73-1P 13227-37-7P 16587-39-6P 16619-97-9P 18929-58-3P  
 33574-65-1P 33574-67-3P 33704-97-1P 38135-10-3P 42894-00-8P  
 52210-82-9P 56608-91-4P 68767-79-3P 68767-83-9P 68767-84-0P  
 68767-85-1P **68767-86-2P**  
 RL: SPN (Synthetic preparation); PREP (Preparation)  
 (preparation of)

IT **68767-86-2P**  
 RL: SPN (Synthetic preparation); PREP (Preparation)  
 (preparation of)

RN 68767-86-2 HCAPLUS

CN Indeno[2,1-a]fluorene, 11,12-dihydro-11,12-diphenyl-, trans- (9CI) (CA INDEX NAME)

Relative stereochemistry.



L96 ANSWER 10 OF 20 HCAPLUS COPYRIGHT 2007 ACS on STN

AN 1972:475056 HCAPLUS

DN 77:75056

OREF 77:12399a,12402a

TI Fluorene acenes and fluorenaphenes. Synthesis of indenofluorenes. XVI.

5-Methyl-, 5,6-dimethyl-, and 5,6-diphenyl-11,12-dihydroindeno[2,1-a]fluorenes

AU Chardonnens, Louis; Bitsch, Serge  
 CS Inst. Chim. Inorg. Anal., Univ. Fribourg, Fribourg, Switz.  
 SO Helvetica Chimica Acta (1972), 55(5), 1345-52  
 CODEN: HCACAV; ISSN: 0018-019X

DT Journal  
 LA French

AB 5-Methyl-11,12-dihydroindeno[2,1-a]fluorene was prepared by condensing PhCH:CMcCH:CHPh with maleic anhydride to give 4-methyl-3,6-diphenyl-1,2,3,6-tetrahydrophthalic anhydride, which was aromatized with S, cyclized with AlCl<sub>3</sub>-NaCl-KCl and reduced with Zn-H. 5,6-Dimethyl-11,12-dihydroindeno[2,1-a]fluorene was similarly prepared from PhCH:CMcCMc:CHPh. 5,6-Diphenyl-11,12-dihydroindeno[2,1-a]fluorene was prepared by reducing its 11,12-dioxo derivative

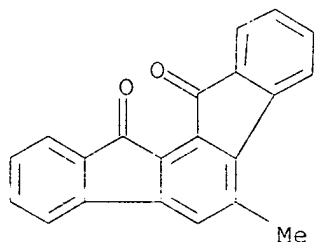
CC 26-4 (Condensed Aromatic Compounds)

IT 1162-64-7P 33776-38-4P 38023-56-2P 38023-57-3P 38023-58-4P  
 38023-59-5P 38023-60-8P **38023-61-9P 38023-62-0P**  
 38023-63-1P 38023-64-2P 38023-65-3P **38023-66-4P**  
**38023-67-5P** 38023-68-6P 38023-69-7P **38023-71-1P**  
**38023-72-2P**  
 RL: SPN (Synthetic preparation); PREP (Preparation)  
 (preparation of)

IT **38023-61-9P 38023-62-0P 38023-66-4P**  
**38023-67-5P 38023-71-1P 38023-72-2P**  
 RL: SPN (Synthetic preparation); PREP (Preparation)  
 (preparation of)

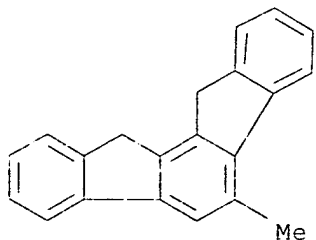
RN 38023-61-9 HCAPLUS

CN Indeno[2,1-a]fluorene-11,12-dione, 5-methyl- (CA INDEX NAME)



RN 38023-62-0 HCAPLUS

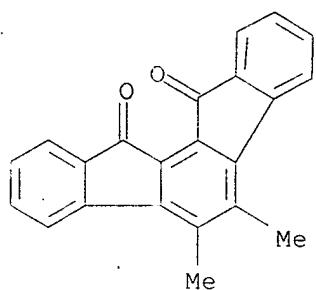
CN Indeno[2,1-a]fluorene, 11,12-dihydro-5-methyl- (CA INDEX NAME)



RN 38023-66-4 HCAPLUS

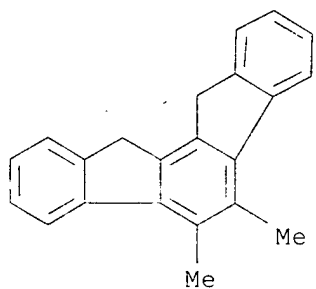
CN Indeno[2,1-a]fluorene-11,12-dione, 5,6-dimethyl- (CA INDEX NAME)





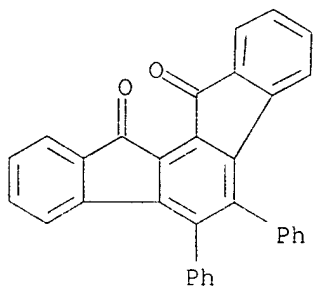
RN 38023-67-5 HCAPLUS

CN Indeno[2,1-a]fluorene, 11,12-dihydro-5,6-dimethyl- (CA INDEX NAME)



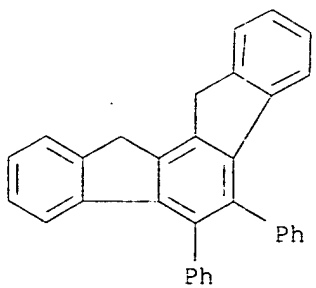
RN 38023-71-1 HCAPLUS

CN Indeno[2,1-a]fluorene-11,12-dione, 5,6-diphenyl- (CA INDEX NAME)

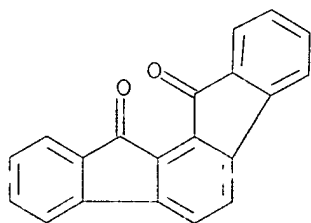


RN 38023-72-2 HCAPLUS

CN Indeno[2,1-a]fluorene, 11,12-dihydro-5,6-diphenyl- (CA INDEX NAME)

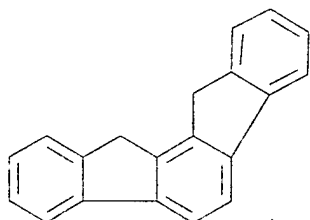


L96 ANSWER 11 OF 20 HCAPLUS COPYRIGHT 2007 ACS on STN  
AN 1965:86263 HCAPLUS  
DN 62:86263  
OREF 62:15399d-e  
TI Column and thin-layer chromatographic separation of polynuclear  
ring-carbonyl compounds  
AU Sawicki, E.; Stanley, T. W.; Elbert, W. C.; Morgan, M.  
CS U.S. Dept. of Health Educ. and Welfare, Cincinnati, OH  
SO Talanta (1965), 12(6), 605-16  
CODEN: TLNTA2; ISSN: 0039-9140  
DT Journal  
LA English  
AB Methods for the separation of polynuclear ring-carbonyl compds. by Al2O3 column  
chromatography and Al2O3 and cellulose thin-layer chromatography are  
described. A method for the thin-layer chromatographic separation of acridones  
and phenanthridones from other types of polynuclear compds. is also  
described. Compds. on a thin-layer chromatogram are located by  
fluorescence methods employing trifluoroacetic acid fumes and Et4NOH solution  
Fluorescence spectra of these compds. on the thin-layer chromatogram and  
in solution also are reported.  
CC 2 (Analytical Chemistry)  
IT **Fluorescence**  
(of carbonyl (polynuclear) compds.)  
IT 82-05-3, 7H-Benz[de]anthracen-7-one 83-33-0, 1-Indanone 90-44-8,  
Anthrone 90-47-1, Xanthen-9-one 91-64-5, Coumarin 134-32-7,  
1-Naphthylamine 486-25-9, Fluoren-9-one 492-22-8, Thioxanthen-9-one  
548-39-0, Phenalen-1-one 578-95-0, 9-Acridanone 604-59-1,  
7,8-Benzoflavone 610-49-1, 1-Anthramine 784-04-3, Ketone, 9-anthryl  
methyl 800-16-8, Ketone, phenyl 2-pyrenyl 1015-89-0,  
6(5H)-Phenanthridinone 1210-35-1, 5H-Dibenzo[a,d]cyclohepten-5-one,  
10,11-dihydro- 2039-77-2, Ketone, methyl 9-phenanthryl 3073-99-2,  
5(12H)-Naphthacenone 3074-00-8, 6H-Benzo[cd]pyren-6-one 3074-03-1,  
11H-Benzo[b]fluoren-11-one 3074-05-3, Ketone, 6-chrysenyl phenyl  
**3074-14-4**, Indeno[2,1-a]fluorene-11,12-dione 3264-21-9, Ketone,  
methyl 1-pyrenyl 3264-24-2; 7H-Dibenzo[c,h]xanthen-7-one  
(chromatography and fluorescence of)  
IT **3074-14-4**, Indeno[2,1-a]fluorene-11,12-dione  
(chromatography and fluorescence of)  
RN 3074-14-4 HCAPLUS  
CN Indeno[2,1-a]fluorene-11,12-dione (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)



L96 ANSWER 12 OF 20 HCAPLUS COPYRIGHT 2007 ACS on STN  
AN 1963:4620C2 HCAPLUS  
DN 59:62002  
OREF 59:11378c-e  
TI 2,2'-Biindene

AU Schroth, W.; Schmidt, K.  
CS Karl-Marx Univ., Leipzig, Germany  
SO Zeitschrift fuer Chemie (1963), 3(8), 309  
CODEN: ZECEAL; ISSN: 0044-2402  
DT Journal  
LA Unavailable  
GI For diagram(s), see printed CA Issue.  
AB The title compound (I) was prepared by the following two methods. (A)  
Addition  
of Br to indene in EtOH gave 1-ethoxy-2-bromoindan, which was converted  
with Mg to 1,1'-diethoxy-2,2'-biindan (II). II heated to about  
100° in the presence of iodine gave 25-30% I, m. 252°,  
fluorescing leaflets (ultraviolet light). (B) Pinacol reduction of  
β-indanone with Al in C<sub>6</sub>H<sub>6</sub> gave III, m. 158°. Dehydration of  
III with Ac<sub>2</sub>O and traces of H<sub>2</sub>SO<sub>4</sub> gave, without rearrangement, 20-25% I.  
I can be used to prepare polycyclic systems. E.g., endo-cis-fluorenaphene  
(IV), m. 285-7°, was prepared via diene addition with maleic anhydride  
(m.p. of the adduct 264-7°), saponification to the free carboxylic acid,  
dehydration with S, and decarboxylation. IV was identical with a product  
prepared in 5-stages from 1,4-diphenylbutadiene (W. Deuschel, CA 45,  
10224e).  
CC 36 (Condensed Aromatic Compounds)  
IT 787-61-1P, 2,2'-Biindene **5815-59-8P**, Indeno[2,1-a]fluorene,  
11,12-dihydro- 93877-16-8P, [2,2'-Biindan]-2,2'-diol 97115-89-4P,  
2,2'-Biindan, 1,1'-diethoxy-  
RL: PREP (Preparation)  
(preparation of)  
IT **5815-59-8P**, Indeno[2,1-a]fluorene, 11,12-dihydro-  
RL: PREP (Preparation)  
(preparation of)  
RN 5815-59-8 HCAPLUS  
CN Indeno[2,1-a]fluorene, 11,12-dihydro- (CA INDEX NAME)



L96 ANSWER 13 OF 20 HCAPLUS COPYRIGHT 2007 ACS on STN  
AN 1960:62624 HCAPLUS  
DN 54:62624  
OREF 54:12084b-i  
TI The pyrolysis of phenanthrene  
AU Lang, Karl Friedrich; Buffleb, Herbert; Kalow, Josef  
CS Rutgerswerke Akt.-Ges., Castrop-Rauxel, Germany  
SO Chemische Berichte (1960), 93, 303-9  
CODEN: CHBEAM; ISSN: 0009-2940  
DT Journal  
LA Unavailable  
OS CASREACT 54:62624  
GI For diagram(s), see printed CA Issue.  
AB The pyrolysis of phenanthrene (I) was investigated. I (1.5 kg.) passed at  
400 g./hr. over porous clay chips in a stainless steel tube at

720-50° and the condensate (1025 g.) fractionated gave 912 g. unchanged I and 105 g. distillation residue at 5 mm. The residue dissolved in hot xylene and cooled gave 6.1 g. crystals, m. 290-5°, and then 0.6 g. crystals, m. 284-7°. The crystalline product sublimed in vacuo up to 400° and the yellow sublimate (5.25 g.) boiled with maleic anhydride (II), diluted with warm xylene, and filtered gave 4.4 g. colorless 2,2'-biphenanthryl (III), m. 306-7.5° (xylene and sublimed). The sublimation residue sublimed in vacuo at 500° and the sublimate recrystd. from boiling pyrene and C10H7Me gave a colorless hydrocarbon,  $\lambda$  338 m $\mu$ . The xylene mother liquor from the crude III heated with 30 g. II to boiling and cooled deposited 1.8 g. IV. The IV treated with excess boiling II and chloranil, diluted with warm xylene, and cooled gave 1.3 g. 1,12:2,3:10,11-tribenzoperylene-1',2'-dicarboxylic acid anhydride (V), m. 406-11° (sublimed). The mother liquor from IV shaken with dilute aqueous NaOH gave at the interphase 0.38 g. VI. The VI acidified and treated with II and chloranil gave V. V and excess soda-lime heated at 350-400°/1 mm. gave a sublimate of 1,12:2,3:10,11-tribenzoperylene, pale yellow needles, m. 381-1.5° (xylene). The mother liquor from the VI dried and chromatographed on Al2O3 gave material containing 20% 2,3:8,9-dibenzoperylene (VII); a 1.5-g. portion, excess II, and chloranil refluxed 5 hrs., cooled, and diluted with xylene gave 0.65 g. red-violet product which fractionally sublimed at 350-450° gave 1,12:2,3:8,9-tribenzoperylene-1',2'-dicarboxylic acid anhydride, red crystals, m. 409-15°, and 1,2:7,8-dibenzocoronene-3,4:9,10-tetracarboxylic acid anhydride (VIII), brown crystals, m. above 480° (PhNO2). VIII decarboxylated in the usual manner and recrystd. from C10H7Me yielded 1,2:7,8-dibenzocoronene (IX), violet in warm concentrated H2SO4. The mother liquor from the chromatogram treated with excess picric acid and the resulting orange-red picrate, m. 216-17.5°, decomposed with NH4OH gave a hydrocarbon, C28H16 (X), yellow felted needles, m. 260-1° (xylene), probably a phenanthrylenephenanthrene. The mother liquor from X fractionally recrystd. yielded a biphenanthrene (XI), m. 175-5.5°, and another biphenanthrene (XII), needles, m. 210-11.5°. The ultraviolet absorption spectra of III, VII, IX, X, XI, XII, di-K 1,2:7,8-dibenzocoronene-5,6-dicarboxylate, tetra-K 1,2:7,8-dibenzocoronene-4,4,9,10-tetracarboxylate, and 2,3:10,11-dibenzoperylene were recorded.

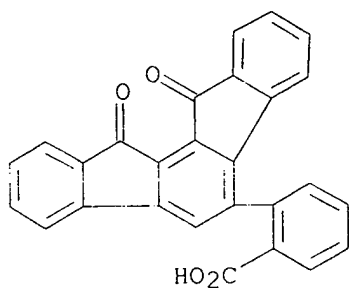
CC 10F (Organic Chemistry: Condensed Carbocyclic Compounds)

IT 190-72-7P, Dibenzo[a,j]coronene 190-81-8P, Tribenzo[b,n,pqr]perylene  
 197-74-0P, Dibenzo[fg,qr]pentacene 4482-09-1P, Dibenzo[a,j]coronene-  
 1,2,9,10-tetracarboxylic 1,2:9,10-dianhydride 4730-60-3P,  
 Tribenzo[b,n,pqr]perylene-15,16-dicarboxylic anhydride 5724-41-4P,  
 Benzo[qr]naphtho[2,1,8,7-fghi]pentacene-1,2-dicarboxylic anhydride  
 121499-69-2P, Tribenzo[b,n,pqr]perylene-15,16-dicarboxylic anhydride,  
 14b,15,16,16a-tetrahydro- 121499-70-5P, Tribenzo[b,n,pqr]perylene-15,16-  
 dicarboxylic acid, 15,16-dihydro-, disodium salt **124121-54-6P**,  
 Benzoic acid, o-(11,12-dihydro-11,12-dioxoindeno[2,1-a]fluoren-5-yl)-  
 RL: (PREP (Preparation)  
 (preparation of)

IT **124121-54-6P**, Benzoic acid, o-(11,12-dihydro-11,12-dioxoindeno[2,1-  
 a]fluoren-5-yl)-  
 RL: PREP (Preparation)  
 (preparation of)

RN 124121-54-6 HCAPLUS

CN Benzoic acid, o-(11,12-dihydro-11,12-dioxoindeno[2,1-a]fluoren-5-yl)-  
 (6CI) (CA INDEX NAME)



L96 ANSWER 14 OF 20 HCAPLUS COPYRIGHT 2007 ACS on STN

AN 1958:11061 HCAPLUS

DN 52:11061

OREF 52:1981c-i,1982a-i,1983a-i,1984a-i,1985a

TI Autoxidation of orthoquinoid indenofluorene hydrocarbons

AU Le Berre, Andre

CS Univ. Paris

SO Ann. chim. (Paris) (1957), 2, 371-425

DT Journal

LA Unavailable

AB Biradicaloid o-quinoid hydrocarbons, 11,12-diphenylideno[2,1-a]fluorene (I) and 13,14-diphenylbenz[c]indeno[2,1-a]fluorene (II), capable of acting as free radicals and undergoing autoxidation, were prepared. Freshly distilled SOCl<sub>2</sub> (50 cc.) and 10 g. 2-phenyl-9-oxo-1-fluorene-carboxylic acid, m. 198-9°, refluxed 45 min., the excess SOCl<sub>2</sub> evaporated in vacuo, the crude chloride heated in 15 min. from 150° to 250°, the crude fluorenedione taken up in 1.2 l. boiling xylene, filtered hot, and the filtrate cooled and filtered gave 8-8.5 g. indeno[2,1-a]fluorene-11,12-dione (III), m. 300-1 (PhNO<sub>2</sub>), λ 257.5, 294, 375, 392, 440-50 mμ (log ε 4.54, 4.57, 3.71, 3.70, 2.68, all measurements in CHCl<sub>3</sub>); 2-phenyl-9-oxo-1-fluorene-carbonyl chloride (IIIa), m. 192-4° (anhydrous C<sub>6</sub>H<sub>6</sub>). Pure III (1 g.) in 25 cc. MeOH treated 30 min. with 0.5 g. KBH<sub>4</sub>, the excess reagent destroyed with 2 cc. AcOH, filtered, and the precipitate washed with a few cc. MeOH gave 0.65 g. 11,12-dihydro-11,12-dihydroxyindeno[2,1-a]fluorene (IV), m. 274-5°, λ 316, 330 mμ (log ε 4.54, 4.47). The MeOH mother liquor poured into H<sub>2</sub>O and filtered gave 0.16 g. of the low-melting isomer (IVa), m. 218-19° (MeOH, after heating at 100°/0.5 mm.), converted into IV by heating several min. at 220°/0.5 mm. IV (or IVa) (25 mg.) in 2 cc. 5% KOH in MeOH boiled several min. and the dark red mixture stirred in air gave 22 mg. III. IV (or IVa) (0.1 g.) boiled 10 min. in 10 cc. 10% HBr in AcOH, the cooled mixture filtered, and the precipitate washed

with AcOH and H<sub>2</sub>O gave 0.13 g. 11,12-dibromo-11,12-dihydroindeno[2,1-a]fluorene (IVb), m. 214-15° (EtOAc), λ 253, 306, 323 (log ε 4.46, 4.29, 4.29). IVb (0.1 g.) treated 24 hrs. with 0.2 g. powdered Zn in 10 cc. AcOH at 20°, the mixture filtered, the residue washed repeatedly with hot C<sub>6</sub>H<sub>6</sub>, the filtrate and washings washed with H<sub>2</sub>O, dried over Na<sub>2</sub>SO<sub>4</sub>, evaporated, and the residue washed with Et<sub>2</sub>O gave 16 g. 11,12-dihydroindeno[2,1-a]fluorene, m. 288-8.5° (xylene), λ 309, 324 mμ (log ε 4.65, 4.68). IV (0.1 g.) heated 10 min. at 275° in a sealed evacuated tube and the dried product crystallized from EtOAc gave 75-80 mg. indeno[2,1-a]fluorene-11(12H)-one (V), m. 215°, λ 288, 440 mμ (log ε 4.84, 3.32), together with a few crystals of 11,12-dihydro-11-hydroxyindeno[2,1-a]fluorene (VI). V (50 mg.) in 2 cc. MeOH treated 30 min. at 20° with 25 mg. KBH<sub>4</sub>,

and the mixture diluted with H<sub>2</sub>O and filtered gave 60 mg. VI, m. 223-5° (solidifying and m. 232-3°),  $\lambda$  255, 311.5 m $\mu$  (log  $\epsilon$  4.06, 4.54). V (50 mg.) and 34 mg. N-bromosuccinimide refluxed 3 hrs. in 5 cc. CCl<sub>4</sub> and the product crystallized from C<sub>6</sub>H<sub>6</sub> yielded 52 mg. 12-Br derivative of V, m. 243-4°,  $\lambda$  260, 294, 440 m $\mu$  (log  $\epsilon$  4.48, 4.56, 3.13). III (5 g.) in 500 cc. dry C<sub>6</sub>H<sub>6</sub> treated with 7.5 g. PhMgBr in 100 cc. Et<sub>2</sub>O (N atmospheric), stored 3 hrs. at 20°, and poured onto ice containing a small volume of HCl, the C<sub>6</sub>H<sub>6</sub> solution washed with 10% HCl and H<sub>2</sub>O, the dried solution evaporated, and the residue chromatographed over Al<sub>2</sub>O<sub>3</sub> in a min. of Cl<sub>2</sub>C:CHCl and eluted with C<sub>6</sub>H<sub>14</sub> and C<sub>6</sub>H<sub>6</sub> gave 5.45 g. 11,12-dihydro-11,12-dihydroxy-11,12-diphenylindeno[2,1-a]fluorene (VII), m. 224° (alc.),  $\lambda$  320, 338 m $\mu$  (log  $\epsilon$  4.56, 4.50), 0.04 g. isomeric compound (VIIa), m. 276° (C<sub>6</sub>H<sub>6</sub>, after heating at 100°/0.5 mm.),  $\lambda$  323, 340.5 m $\mu$  (log  $\epsilon$  4.50, 4.46), and 0.11 g. 12-hydroxy-phenylindeno[2,1-a]fluorene-11(12H)-one, m. 227-8° (C<sub>6</sub>H<sub>6</sub>, after heating at 100° 0.5 mm.),  $\lambda$  253, 298, 450 m $\mu$  (log  $\epsilon$  4.50, 4.62, 3.25), converted with PhMgBr to VII. VII (25 mg.) treated 1 hr. at 20° with 0.25 cc. Ac<sub>2</sub>O containing a trace of ZnCl<sub>2</sub> and the product washed with AcOH gave 15 mg. 11,12-diacetate of VII, m. 309-10°,  $\lambda$  320.5, 338.5 m $\mu$  (log  $\epsilon$  4.58, 4.52). VII (1 g.) boiled 10 min. with 25 cc. 10% HBr in AcOH with stirring, the cooled mixture filtered, and the crystalline precipitate washed with AcOH and H<sub>2</sub>O gave 1.18 g. crude product, m. 260-2°, which, taken up in a min. of CHCl<sub>4</sub> and precipitated with 2 vols. Et<sub>2</sub>O yielded 1 g. 11,12-dibromo-11,12-dihydro-11,12-diphenylindeno[2,1-a]fluorene (VIII), m. 263-6° (decomposition),  $\lambda$  264, 330 m $\mu$  (log  $\epsilon$  4.49, 4.25). VIII (0.1 g.) in 5 cc. warm dioxane diluted with 5 cc. H<sub>2</sub>O, stored, diluted with 5 cc. H<sub>2</sub>O, and the crude product separated by fractional crystallization from alc. yielded 52 mg. VII and 19 mg. VIIa. VII (0.25 g.), 0.25 g. KI, and 0.3 g. NaH<sub>2</sub>PO<sub>2</sub>·H<sub>2</sub>O refluxed 3 hrs. in 25 cc. AcOH, the solution poured into aqueous Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>, filtered, the precipitate taken up in C<sub>6</sub>H<sub>6</sub>, the solution washed with H<sub>2</sub>O, the extract evaporated, and the residue fractionally crystallized from AcOH gave 25 mg. 11,12-dihydro-11,12-diphenylindeno[2,1-a]fluorene (IX), m. 328-30° (C<sub>6</sub>H<sub>6</sub>),  $\lambda$  315.5, 331 m $\mu$  (log  $\epsilon$  4.60, 4.64), and 0.16 g. isomeric compound (IXa), m. 266° (C<sub>6</sub>H<sub>6</sub>),  $\lambda$  312, 327 m $\mu$  (log  $\epsilon$  4.64, 4.68). VIII (0.25 g.) boiled 10 min. with 0.5 g. powdered Zn in 25 cc. AcOH and the crude product washed with C<sub>6</sub>H<sub>14</sub> and fractionated in AcOH gave 27 mg. IX and 0.1 g. di-AcO derivative, m. 309-10°. VIII (1 g.) in 50 cc. C<sub>6</sub>H<sub>6</sub> refluxed 30 min. with stirring in the presence of 0.5 g. active Cu [cf. Piccard, Helv. Chim. Acta 5, 147(1922)] (all operations in a CO<sub>2</sub> atmospheric), the cold solution filtered, the residual Cu washed several times with C<sub>6</sub>H<sub>6</sub>, the exts. and filtrate evaporated in vacuo, the crude product taken up in 50 cc. boiling EtOAc, the solution concentrated to about 20 cc., cooled slowly, the mother liquors decanted, and the crystals washed with EtOAc and Et<sub>2</sub>O and dried immediately in vacuo gave 0.4-0.5 g. violet-black crystalline I, m. 249-51°,  $\lambda$  287, 556 m $\mu$  (log  $\epsilon$  4.62, 4.03), converted by Br in CCl<sub>4</sub> to VIII. I (0.25 g.) boiled 15 min. with 0.5 g. powdered Zn in 30 cc. AcOH (N atmospheric), and the product washed with Et<sub>2</sub>O and fractionated in C<sub>6</sub>H<sub>6</sub> and AcOH gave 0.13 g. IX and 0.07 g. IXa. I (0.25 g.) boiled 1.5 hrs. (N atmospheric) with 0.25 g. KBH<sub>4</sub> in 25 cc. alc. and 25 cc. tetrahydrofuran, the solution poured into H<sub>2</sub>O,

the precipitate taken up in C6H6, the extract evaporated, and the residue washed with

Et2O gave 0.2 g. IXa. I (50 mg.) and 15 mg. maleic anhydride in 1 cc. xylene boiled 50 hrs. in an evacuated sealed tube, the solution cooled and filtered, the precipitate washed with Et2O, and the product (44 mg.) recrystd. from C6H6 gave the adduct, C36H22O3, m. 315°,  $\lambda$  321, 337.5

$\mu$  (log  $\epsilon$  4.47, 4.46). A series of benz[c]indeno[2,1-a]fluorene derivs. were prepared. The maleic anhydride adduct of diphenylisobenzofuran (5 g.) treated 40-50 hrs. with 100 cc. pure H2SO4 according to the procedure of Weiss and Abel's (C.A. 27, 722), the green liquid poured onto cracked ice, filtered, and the precipitate washed with hot H2O, dried, washed with hot C6H6, and crystallized from xylene or PhNO2 gave 2.5-3.0 g. benz[c]indeno[2,1-a]fluorene-13,14-dione (X), m.

309-10°,  $\lambda$  265, 290, 440-50  $\mu$  (log  $\epsilon$  4.79, 4.57, 3.76). X (1 g.) in 200 cc. MeOH and 200 cc. CHCl3 stirred gently 30 min. to 2 hrs. with 0.5 g. KBH4 to complete solution, treated with 10 cc. AcOH, stored overnight, filtered, and the crystalline precipitate washed with MeOH

gave 0.87

g. 13,14-dihydro-13,14-dihydroxybenz[c]indeno[2,1-a]fluorene (XI), m. 309-12°,  $\lambda$  253, 369, 388  $\mu$  (log  $\epsilon$  4.58, 4.43, 4.39). XI (50 mg.) and 3 g. C10H8 boiled 3 hrs. and the excess C10H8 extracted with Et2O gave 26 mg. X, similarly obtained by 3 hrs. boiling in PhNO2, by fusion in air, and by autoxidation in alkaline media. XI (0.1 g.) heated 10 min. in 10 cc. 10% HBr in AcOH gave 0.11 g. 13,14 - dibromo - 13,14 - dihydrobenz[c]indeno[2,1 - a]fluorene (XII), m. 315-18° (dioxane),  $\lambda$  264, 319, 375, 390  $\mu$  (log  $\epsilon$  4.77, 3.98, 4.21, 4.17). XII with active Cu gave only amorphous resins. XII (0.1 g.) boiled 15 min. in 10 cc. AcOH with 0.2 g. powdered Zn and the product

crystallized

from EtOAc and xylene yielded 30% 13,14-dihydrobenz[c]indeno[2,1-a]fluorene (XIII), m. 218-19°,  $\lambda$  249, 355, 373  $\mu$  (log  $\epsilon$  4.50, 4.54, 4.53). XI (0.25 g.) and 8 g. tech. C10H8 heated 5 hrs. at 250-60° in a sealed evacuated tube, the cooled melt extracted with Et2O, and the residue crystallized from xylene gave 0.15-0.16 g. benz[c]indeno[2,1-a]fluorene-13(14H)-one (XIV), m. 255°,  $\lambda$  242, 277, 310, 355, 483  $\mu$  (log  $\epsilon$  4.55, 4.52, 4.52, 3.92, 3.54), oxidized by refluxing 12 hrs. with 2 parts by weight SeO2 in 20 parts by volume AcOH to 65% X. XIV (0.1 g.) in 20 cc. EtOH and 20 cc. CHCl3 boiled 15-20 min. with 0.05 g. KBH4, the colorless solution treated with 2 cc. AcOH and the cooled solution filtered, and the crystals washed with alc. and dried gave 90% 12,14-dihydro-13-hydroxybenz[c]indeno[2,1-a]fluorene, m. 263-5° (dioxane),  $\lambda$  253, 364, 382  $\mu$  (log  $\epsilon$  4.45, 4.33, 4.32), converted by 10% HBr in AcOH to a yellow crystalline mixture of 13-bromobenz[c]indeno[2,1-a]fluorene with some XII, m. 243-6° (xylene); reduced by boiling 3 hrs. with powdered Zn in AcOH to XIII. XIV (0.1 g.) and 0.06 g. N-bromosuccinimide boiled 1 hr. in 10 cc. CCl4 and the product washed with Et2O gave 0.1 g. 14-Br derivative of XIV, m. 252-3°,  $\lambda$  258, 320, 377, 480  $\mu$  (log  $\epsilon$  4.66, 4.28, 3.84, 3.47). Phenylation of 5 g. X in 500 cc. C6H6 with 5.5 g. PhMgBr in 100 cc. Et2O and chromatography of the product in Cl2C:CHCl on Al2O3 yielded 4.45 g. 13,14-dihydro-13,14-dihydroxy-13,14-diphenylbenz[c]indeno[2,1-a]fluorene (XV), m. 299-300° (showing dimorphism and thermochromy),  $\lambda$  244, 255, 357, 374, 394  $\mu$  (log  $\epsilon$  4.62, 4.60, 4.20, 4.43, 4.43), 0.060 g. isomeric compound (XVa), m. 348-9° (thermochromic),  $\lambda$  243, 252.5, 358.5, 376, 396.5  $\mu$  (log  $\epsilon$  4.64, 4.62, 4.20, 4.43, 4.42), and 0.28 g. 14-hydroxy-14-phenylbenz[c]indeno[2,1-a]fluorene-13(14H)-one, m. 289-90°,  $\lambda$  252.5, 320, 361, 500  $\mu$  (log  $\epsilon$  4.63, 4.42, 3.99, 3.47), further phenylated with PhMgBr to XV. XV (or XVa) (1 g.) refluxed 10 min. in 25 cc. 10% HBr in AcOH, filtered, and the yellow

cryst, precipitate washed with a small amount of AcOH and with H<sub>2</sub>O gave 1.00 g. 13,14-dibromo-13,14-dihydro-13,14-diphenylbenz[c]indeno[2,1-a]fluorene (XVI), m. 254-6° (decomposition),  $\lambda$  265, 385, 400 m $\mu$  (log  $\epsilon$  4.72, 4.17, 4.14). XV (0.25 g.), 0.4 g. KI, and 0.8 g. NaH<sub>2</sub>PO<sub>2</sub>·H<sub>2</sub>O in 25 cc. AcOH refluxed 8 hrs. and poured into aqueous Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>, the mixture filtered, the precipitate taken up in C<sub>6</sub>H<sub>6</sub>, the extract evaporated, and the stereoisomeric mixture separated by chromatography from C<sub>6</sub>H<sub>14</sub> on Al<sub>2</sub>O<sub>3</sub> yielded 0.16 g. 13,14-dihydro-13,14-diphenylbenz[c]indeno[2,1-a]fluorene, m. 281-2° (EtOAc),  $\lambda$  252, 342-348, 358.5, 377.5 m $\mu$  (log  $\epsilon$  4.57, 4.28, 4.50, 4.49), and 25 mg. of an isomer, m. 292-3° (C<sub>6</sub>H<sub>6</sub>),  $\lambda$  248, 345-352, 364, 382.5 m $\mu$  (log  $\epsilon$  4.57, 4.25-4.26, 4.46, 4.44). The same isomers were obtained by treating 0.5 g. XVI 30 min. with 2 g. powdered Zn in 50 cc. AcOH at 20°. Attempts to obtain II from XVI with metals in organic solvents in vacuo or under inert gases gave solns., at first intensely green and later yellowish brown with a blue-violet fluorescence, yielding only resinous products on evaporation XVI (0.5 g.) and 0.085 g. maleic anhydride in 25 cc. dry C<sub>6</sub>H<sub>6</sub> treated 1 hr. with 300 mg. active Cu at 20° (N atmospheric), filtered, the Cu residue washed with hot C<sub>6</sub>H<sub>6</sub>, the filtrate and washings evaporated, and the residue washed with Et<sub>2</sub>O and crystallized from xylene gave 49 mg. adduct, C<sub>40</sub>H<sub>20</sub>O<sub>3</sub>, m. 366-8°,  $\lambda$  261, 386 m $\mu$  (log  $\epsilon$  4.36, 4.40). In neutral solvents (Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, CHCl<sub>3</sub>, CS<sub>2</sub>) with or without addition of antioxidants [4,2,6-Me(tert-Bu)<sub>2</sub>C<sub>6</sub>H<sub>2</sub>OH (XVIa), hydroquinone (XVIb), pyrogallol (XVIc)] or in the presence of AcOH, I rapidly adsorbed O with change of color from blue-violet to pale yellow with or without irradiation. Autoxidation in C<sub>6</sub>H<sub>6</sub>, CS<sub>2</sub>, or CHCl<sub>3</sub> without additives but under all conditions of temperature, concentration, irradiation, or lack of irradiation, and evaporation of the solvent in vacuo at 20° gave a pale beige amorphous powder, C<sub>32</sub>H<sub>20</sub>O<sub>2</sub>, reduced by LiAlH<sub>4</sub> in tetrahydrofuran to a small amount of an isomer of 9-hydroxy-2-(o-hydroxyphenyl)-9-phenyl-1-( $\alpha$ -hydroxybenzyl)fluorene (XVII), m. 240°. Autoxidation in Et<sub>2</sub>O and in CS<sub>2</sub> gave the same powder, together with traces of VIIa and 1-benzoyl-9-hydroxy-2-(o-hydroxyphenyl)-9-phenylfluorene (XVIII), m. 266-8°, resp. Autoxidation of 50 mg. I in C<sub>6</sub>H<sub>6</sub> in the presence of 2 mg. XVIa, XVIb, or XVIc gave resins containing 0, 13, or 74% VIIa. I (50 mg.) in 10 cc. C<sub>6</sub>H<sub>6</sub> containing 0.1, 1.0, and 2.0 cc. AcOH, resp., gave on autoxidation, 0, 49, and 57% XVIII. I (0.2 g.) in 40 cc. dioxane and 20 cc. AcOH decolorized by a slow current of O, the pale yellow solution poured into H<sub>2</sub>O, the mixture filtered, the precipitate washed with H<sub>2</sub>O, recrystd. from xylene, and the crystals desolvated by washing with Et<sub>2</sub>O or by heating yielded 62-6% XVIII, m. 271°,  $\lambda$  243, 287, 328, 342 m $\mu$  (log  $\epsilon$  4.39, 4.29, 4.06, 4.03), also obtained by treatment of VIII with active Cu in C<sub>6</sub>H<sub>6</sub>, chromatographic purification of the product from dioxane on Al<sub>2</sub>O<sub>3</sub>, and elution with C<sub>6</sub>H<sub>14</sub> and EtOH. XVIII (0.1 g.) in 2 cc. anhydrous tetrahydrofuran treated rapidly with 0.05 g. LiAlH<sub>4</sub> added portionwise, the mixture refluxed 4-5 min., the solution separated from the excess LiAlH<sub>4</sub> poured into 100 cc. Et<sub>2</sub>O, the excess LiAlH<sub>4</sub> washed with a small volume of tetrahydrofuran, the washings and Et<sub>2</sub>O solution filtered, the filtrate washed with 10% HCl and H<sub>2</sub>O, evaporated, and the residue recrystd. from EtOH and dried at 140/0.5 mm. gave 9-hydroxy-1-(o-hydroxyphenyl)-9-phenyl-9-( $\alpha$ -hydroxybenzyl)fluorene, m. 205-6°,  $\lambda$  245, 288 m $\mu$  (log  $\epsilon$  4.30, 4.27). XVIII (0.1 g.) in 2 cc. freshly distilled SOCl<sub>2</sub> containing 0.1 g. rigorously dry Na<sub>2</sub>CO<sub>3</sub> kept 30 min. at 20°, the SOCl<sub>2</sub> evaporated in vacuo, the residue extracted with boiling Et<sub>2</sub>O, the extract concentrated, filtered, the product taken up in 15-18 cc. boiling C<sub>6</sub>H<sub>6</sub>, and the extract



concentrated gave 84 mg. 1-benzoyl-9-chloro-2-(o-hydroxyphenyl)-9-phenylfluorene, m. 282-4°,  $\lambda$  250, 293  $\mu$ m (log  $\epsilon$  4.42, 4.19), converted by boiling 20 mg. 30 min. in 5 cc. dioxane diluted with 5 cc. H<sub>2</sub>O, extracting the cooled solution with Et<sub>2</sub>O, and crystallizing the product from xylene to 16 mg. XVIII. XVIII (0.1 g.) treated 45 min. at 20° with 10 cc. 20% HI in AcOH, the mixture poured into aqueous Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>, the precipitate taken up in Et<sub>2</sub>O, the extract evaporated, the mixed product taken up in hot C<sub>6</sub>H<sub>6</sub>, the cooled solution filtered, and the crystalline precipitate washed with EtOAc gave 58 mg. 1,13-diphenyl-13H-1,2-benzopyrano[3,4-a]fluorene (XIX), m. 294-5° (from C<sub>6</sub>H<sub>6</sub>),  $\lambda$  242, 294.5, 326.5  $\mu$ m (log  $\epsilon$  4.33, 4.27, 4.48). The filtrate and washings evaporated, the residue taken up in hot EtOAc, the cooled mixture filtered from 10 mg. mineral product, m. 116-17° (resolidifying and m. 120-1°), and the filtrate cooled and filtered gave 22 mg. isomeric compound (XIXa), m. 226-8°,  $\lambda$  242.5, 292, 324  $\mu$ m (log  $\epsilon$  4.19, 4.25, 4.44). The isomers, XIX and XIXa, were insol. in pure H<sub>2</sub>SO<sub>4</sub> and in KOH in MeOH. IIIa (0.5 g.) in 250 cc. anhydrous Et<sub>2</sub>O treated 1 hr. with 0.6 g. PhMgBr in Et<sub>2</sub>O at 20° (N atmospheric), poured onto ice and HCl, the Et<sub>2</sub>O layer washed with aqueous Na<sub>2</sub>CO<sub>3</sub> and H<sub>2</sub>O, the dried extract evaporated, and the product washed with Et<sub>2</sub>O gave 0.22 g. 1-benzoyl-9-hydroxy-2,9-diphenylfluorene, m. 239° (EtOAc),  $\lambda$  244, 291, 334  $\mu$ m (log  $\epsilon$  4.43, 4.40, 3.70), converted by LiAlH<sub>4</sub> reduction in tetrahydrofuran, crystallization of the product from EtOH and desolvation at 120°/0.5 mm. to 75% 9-hydroxy-2,9-diphenyl-1-( $\alpha$ -hydroxybenzyl)fluorene, m. 180-1°,  $\lambda$  244, 288  $\mu$ m (log  $\epsilon$  4.35, 4.29). II was extremely sensitive to air. XVI (0.5 g.) in 50 cc. C<sub>6</sub>H<sub>6</sub> and 5 cc. AcOH treated 1 hr. at 20° in the presence of air with 0.5 g. active Cu, the mixture filtered, the washed filtrate evaporated, and the resinous product washed with Et<sub>2</sub>O and crystallized repeatedly from xylene yielded 29% 7-hydroxy-5-(o-hydroxyphenyl)-6-benzoyl-7H-benzo[c]fluorene (XX), m. 318-19° (xylene),  $\lambda$  245, 338, 365  $\mu$ m (log  $\epsilon$  4.67, 4.12, 3.92), soluble in 5% KOH in MeOH, and a small amount of the diol XVa. VIII (1 g.) in 100 cc. dioxane at 0° containing 1 g. Na<sub>2</sub>CO<sub>3</sub> treated rapidly dropwise with 20 cc. 80% H<sub>2</sub>O<sub>2</sub>, the mixture stirred 30 min. at 50°, the colorless solution poured into H<sub>2</sub>O, filtered, the precipitate taken up in Et<sub>2</sub>O, the washed and dried extract evaporated in vacuo, and the residue crystallized from Et<sub>2</sub>O gave 0.54 g. 11,12-dihydro-11,12-diphenylindeno[2,1-a]fluorene 11,12-bis(hydroperoxide) (XXI), m. 260-2° (C<sub>6</sub>H<sub>6</sub> and dried at 80°/0.5 mm.),  $\lambda$  240.5, 322.5, 340  $\mu$ m (log  $\epsilon$  4.52, 4.53, 4.45). The Et<sub>2</sub>O mother liquors concentrated to 1 cc., diluted with 1 cc. C<sub>6</sub>H<sub>12</sub>, filtered, and the crystals washed with C<sub>6</sub>H<sub>12</sub> and Et<sub>2</sub>O, recrystd. from C<sub>6</sub>H<sub>6</sub>, and dried at 80°/0.5 mm. gave 47 mg. isomer (XXIa), m. 232-4°,  $\lambda$  242, 320, 337.5  $\mu$ m (log  $\epsilon$  4.52, 4.57, 4.47). XXI (0.1 g.) heated 10 min. at 200°/0.1 mm. evolved 2.30 cc. gas (19°, 760 mm.) containing 13% CO<sub>2</sub> and 83% O; the residue crystallized from CHCl<sub>3</sub>-C<sub>6</sub>H<sub>12</sub> yielded 35% VIIa but no III. XXI (0.125 g.) in 5 cc. warm dioxane cooled and treated 30 min. with 5 cc. 0.1M Pb(OAc)<sub>4</sub> in AcOH, the mixture poured into Et<sub>2</sub>O, the solution washed with 5% NaOH, 10% HCl, and H<sub>2</sub>O, the solvent evaporated, and the resinous product crystallized from Et<sub>2</sub>O-C<sub>6</sub>H<sub>12</sub> and xylene gave 45 mg. XVIII. XXI (0.1 g.) in 50 cc. AcOH containing 0.1 cc. pure H<sub>2</sub>SO<sub>4</sub> kept 24 hrs. at 20°, poured into H<sub>2</sub>O, the precipitate taken up in Et<sub>2</sub>O, the washed and dried solution evaporated, and the residue crystallized from C<sub>6</sub>H<sub>6</sub>

and xylene gave 32 mg. XVIII. XVI (0.5 g.) in 50 cc. dioxane containing 0.25 g. Na<sub>2</sub>CO<sub>3</sub> at 0° treated with 80% H<sub>2</sub>O, the mixture stirred gently 24 hrs. at 20°, poured into excess H<sub>2</sub>O, filtered, the precipitate taken up in Et<sub>2</sub>O, the washed and dried extract evaporated, and the residue crystallized from

boiling Et<sub>2</sub>O gave 0.41 g. 13,14-dihydro-13,14-diphenylbenz[c]indeno[2,1-a]fluorene bis(hydroperoxide) (XXII), m. 180° (foaming),  $\lambda$  244, 360, 378, 398.5 m $\mu$  (log  $\epsilon$  4.64, 4.18, 4.39, 4.38), transformed on boiling in AcOH into XVa. XXII (0.1 g.) heated at 145-50°/0.1 mm. evolved 2.20 cc. gas (25°/760 mm.) containing 36% CO<sub>2</sub> and 61% O; recrystn. of the residue from CHCl<sub>3</sub>-C<sub>6</sub>H<sub>6</sub> gave 17 mg. XVa. No PhOH or XI were formed.

XXII (0.28 g.) in 15 cc. dioxane treated 15 min. at 20° with 15 cc. 0.1M Pb(OAc)<sub>4</sub> in AcOH, the mixture poured into Et<sub>2</sub>O, the solution washed with 5% NaOH, 10% HCl, and H<sub>2</sub>O, the dried extract evaporated, and the residue crystallized

from CHCl<sub>3</sub>-C<sub>5</sub>H<sub>12</sub> and xylene yielded 20% XX. XXII in AcOH containing a trace of H<sub>2</sub>SO<sub>4</sub> rapidly deepened in color from violet-rose to clear violet-red but no crystalline product could be isolated. Although the expected cyclic peroxides were not isolated, their intermediate occurrence as precursors in the formation of the HO ketone end products was confirmed by the results of autoxidation in acid media. Detailed schemes of the stepwise transformations were charted and interpretations of the various possible mechanisms, as supported by ultraviolet absorption measurements, were discussed.

CC 10 (Organic Chemistry)

IT **Fluorescence**

Ultraviolet and visible, spectra  
(of indenofluorene derivs.)

IT **3074-14-4P**, Indeno[2,1-a]fluorene-11,12-dione **5815-59-8P**, Indeno[2,1-a]fluorene, 11,12-dihydro- 34706-87-1P, Benz[c]indeno[2,1-a]fluorene-13,14-dione 102170-95-6P, Fluorene-1-carbonyl chloride, 9-oxo-2-phenyl- 103266-74-6P, Ketone, 9-chloro-2-(o-hydroxyphenyl)-9-phenylfluorene-1-yl phenyl 103266-98-4P, Ketone, 9-hydroxy-2,9-diphenylfluorene-1-yl phenyl 103398-18-1P, Fluorene-1-methanol, 9-hydroxy- $\alpha$ ,2,9-triphenyl- **111032-51-0P**, Indeno[2,1-a]fluorene, 11,12-dibromo-11,12-dihydro- **111386-76-6P**, Indeno[2,1-a]fluorene-11(12H)-one, 12-bromo- **112553-53-4P**, Indeno[2,1-a]fluorene-11(12H)-one 114353-12-7P, Benz[c]indeno[2,1-a]fluorene-13-ol, 13,14-dihydro- 114793-43-0P, Benz[c]indeno[2,1-a]fluorene-13,14-diol, 13,14-dihydro- **114889-39-3P**, Indeno[2,1-a]fluorene-11-ol, 11,12-dihydro- 119925-92-7P, Benz[c]indeno[2,1-a]fluorene-13(14H)-one, 14-hydroxy-14-phenyl- 120233-37-6P, Benz[c]indeno[2,1-a]fluorene-13(14H)-one, 14-bromo- 120233-41-2P, Benz[c]indeno[2,1-a]fluorene, 13,14-dibromo-13,14-dihydro- 120233-49-0P, Benz[c]indeno[2,1-a]fluorene-13(14H)-one 120233-54-7P, Benz[c]indeno[2,1-a]fluorene, 13-bromo-13,14-dihydro- 120233-63-8P, Benz[c]indeno[2,1-a]fluorene, 13,14-dihydro- **120267-45-0P**, Indeno[2,1-a]fluorene, 11,12-dibromo-11,12-dihydro-11,12-diphenyl- **121967-72-4P**, Indeno[2,1-a]fluorene-11,12-diol, 11,12-dihydro-11,12-diphenyl-, diacetate 122273-24-9P, Benz[a]indeno[1,2,3-fg]aceanthrylene-9,10-dicarboxylic anhydride, 8b,9,10,10a-tetrahydro-8b,10a-diphenyl- 124269-74-5P, Hydroperoxide, (13,14-dihydro-13,14-diphenylbenz[c]indeno[2,1-a]fluorene-13,14-ylene)di- 124290-45-5P, Indeno[1,2,3-cd]fluoranthene-5,6-dicarboxylic anhydride, 4b,5,6,6a-tetrahydro-4b,6a-diphenyl- 124513-91-3P, Benz[c]indeno[2,1-a]fluorene, 13,14-dibromo-13,14-dihydro-13,14-diphenyl- 124514-15-4P, Ketone, 7-hydroxy-5-(o-hydroxyphenyl)-7-phenyl-7H-benzo[c]fluorene-6-yl phenyl **876504-70-0P**, Indeno[2,1-a]fluorene-11(12H)-one, 12-hydroxy-12-phenyl-

RL: PREP (Preparation)  
(preparation of)

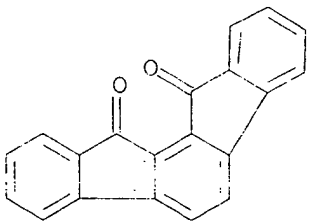
IT 103267-02-3, Ketone, 9-hydroxy-2-(o-hydroxyphenyl)-9-phenylfluoren-1-yl  
phenyl 116032-16-7, Fluorene-1-methanol, 9-hydroxy-2-(o-hydroxyphenyl)-  
 $\alpha$ ,9-diphenyl- 124139-88-4, Benzo[b]fluoreno[2,1-d]pyran,  
11,12-dihydro-11,12-diphenyl- **124145-09-1**, Hydroperoxide,  
(11,12-dihydro-11,12-diphenylindeno[2,1-a]fluoren-11,12-ylene)di-  
**124180-22-9**, Indeno[2,1-a]fluorene-11,12-diol,  
11,12-dihydro-11,12-diphenyl- **124180-97-8**, Indeno[2,1-  
a]fluorene, 11,12-dihydro-11,12-diphenyl- **856642-59-6**,  
Indeno[2,1-a]fluorene-11,12-diol, 11,12-dihydro-  
(stereoisomers)

IT **3074-14-4P**, Indeno[2,1-a]fluorene-11,12-dione **5815-59-8P**  
, Indeno[2,1-a]fluorene, 11,12-dihydro- **111032-51-0P**,  
Indeno[2,1-a]fluorene, 11,12-dibromo-11,12-dihydro- **111386-76-6P**  
, Indeno[2,1-a]fluoren-11(12H)-one, 12-bromo- **112553-53-4P**,  
Indeno[2,1-a]fluoren-11(12H)-one **114889-39-3P**,  
Indeno[2,1-a]fluoren-11-ol, 11,12-dihydro- **120267-45-0P**,  
Indeno[2,1-a]fluorene, 11,12-dibromo-11,12-dihydro-11,12-diphenyl-  
**121967-72-4P**, Indeno[2,1-a]fluorene-11,12-diol,  
11,12-dihydro-11,12-diphenyl-, diacetate **876504-70-0P**,  
Indeno[2,1-a]fluoren-11(12H)-one, 12-hydroxy-12-phenyl-

RL: PREP (Preparation)  
(preparation of)

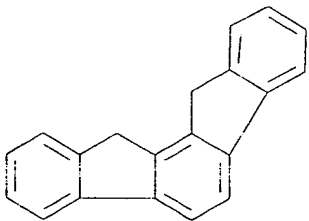
RN 3074-14-4 HCAPLUS

CN Indeno[2,1-a]fluorene-11,12-dione (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)



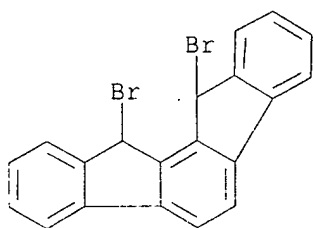
RN 5815-59-8 HCAPLUS

CN Indeno[2,1-a]fluorene, 11,12-dihydro- (CA INDEX NAME)

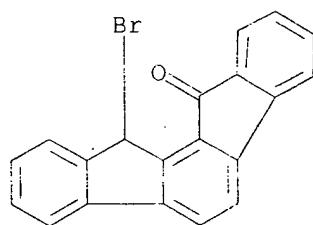


RN 111032-51-0 HCAPLUS

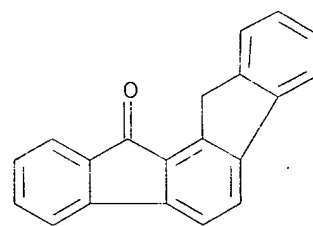
CN Indeno[2,1-a]fluorene, 11,12-dibromo-11,12-dihydro- (CA INDEX NAME)



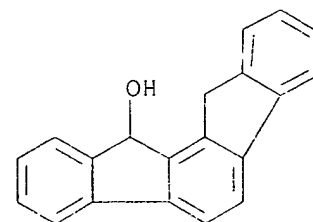
RN 111386-76-6 HCAPLUS  
 CN Indeno[2,1-a]fluoren-11(12H)-one, 12-bromo- (CA INDEX NAME)



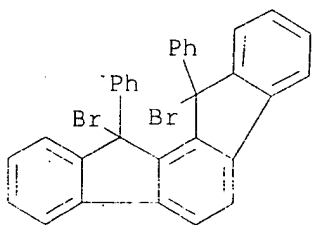
RN 112553-53-4 HCAPLUS  
 CN Indeno[2,1-a]fluoren-11(12H)-one (CA INDEX NAME)



RN 114889-39-3 HCAPLUS  
 CN Indeno[2,1-a]fluoren-11-ol, 11,12-dihydro- (CA INDEX NAME)

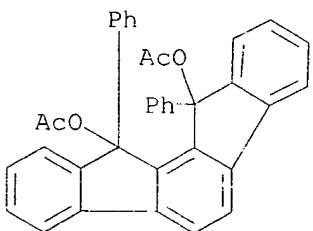


RN 120267-45-0 HCAPLUS  
 CN Indeno[2,1-a]fluorene, 11,12-dibromo-11,12-dihydro-11,12-diphenyl- (CA INDEX NAME)



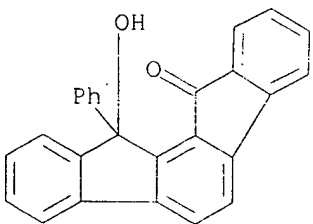
RN 121967-72-4 HCAPLUS

CN Indeno[2,1-a]fluorene-11,12-diol, 11,12-dihydro-11,12-diphenyl-, diacetate (6CI) (CA INDEX NAME)



RN 876504-70-0 HCAPLUS

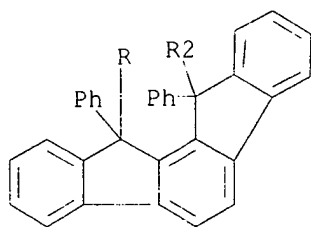
CN Indeno[2,1-a]fluoren-11(12H)-one, 12-hydroxy-12-phenyl- (CA INDEX NAME)



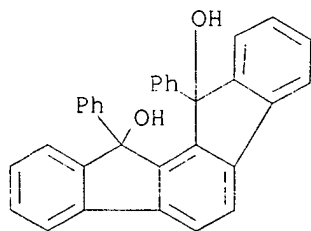
IT **124145-09-1**, Hydroperoxide, (11,12-dihydro-11,12-diphenylindeno[2,1-a]fluoren-11,12-ylene)di- **124180-22-9**, Indeno[2,1-a]fluorene-11,12-diol, 11,12-dihydro-11,12-diphenyl- **124180-97-8**, Indeno[2,1-a]fluorene, 11,12-dihydro-11,12-diphenyl- **856642-59-6**, Indeno[2,1-a]fluorene-11,12-diol, 11,12-dihydro- (stereoisomers)

RN 124145-09-1 HCAPLUS

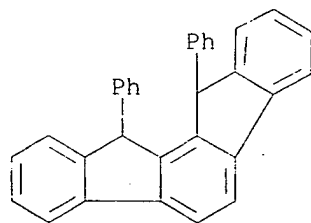
CN Hydroperoxide, (11,12-dihydro-11,12-diphenylindeno[2,1-a]fluoren-11,12-ylene)di- (6CI) (CA INDEX NAME)



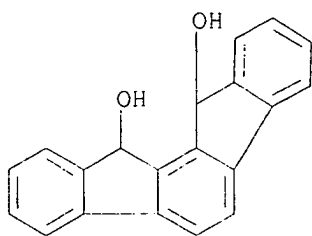
RN 124180-22-9 HCAPLUS  
 CN Indeno[2,1-a]fluorene-11,12-diol, 11,12-dihydro-11,12-diphenyl- (CA INDEX NAME)



RN 124180-97-8 HCAPLUS  
 CN Indeno[2,1-a]fluorene, 11,12-dihydro-11,12-diphenyl- (CA INDEX NAME)



RN 856642-59-6 HCAPLUS  
 CN Indeno[2,1-a]fluorene-11,12-diol, 11,12-dihydro- (CA INDEX NAME)



L96 ANSWER 15 OF 20 HCAPLUS COPYRIGHT 2007 ACS on STN

AN 1957:39173 HCAPLUS

DN 51:39173

OREF 51:7334h-i,7335a-d

TI Indenofluorene o-quinoid hydrocarbons

AU Etienne, Andre; Le Berre, Andre

SO Compt. rend. (1956), 242, 1493-6

DT Journal

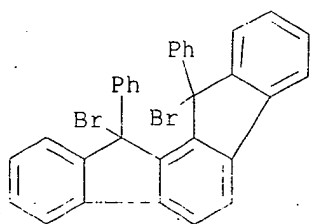
LA Unavailable

AB The reactions of 11,12-diphenylindeno [2,1-a] fluorene (I) and 13,14-diphenylbenz[c]indeno [2,1-a]fluorene (II) were studied to elucidate their structures. Indeno[2,1-a]fluorene-11,12-dione (III) with PhMgBr gave the 2 stereoisomeric 11,12-diphenyl-11,12-dihydroxy-11,12-dihydroindeno [2,1-a]fluorenes (IV), colorless rods, m. 224° (chiefly), and 276°, and a small amount of 11-phenyl-11-hydroxyindeno[2,1-a]fluorene-12-one, yellow-orange rods, m. 227°, separable by chromatography on alumina. Reduction of IV with KIHOAc gave the 2 stereoisomeric 11,12-diphenyl-11,12-dihydroindeno [2,1-a]fluorenes (V), colorless prisms, m. 266°, and colorless needles, m. 330°, separable by differential solubility in HOAc. HBr-HOAc and IV gave 1 stereochem. form of 11,12-diphenyl-11,12-dibromo-11,12-dihydroindeno[2,1-a]-fluorene (VI), yellow prisms, m. 263-6°, hydrolyzed in boiling dioxane-H<sub>2</sub>O to a mixture of IV, reducible with ZnHOAc to isomers m. higher than V and a notable amount of the corresponding diacetate, colorless prisms, m. 310°. VI with specially prepared Cu [Piccard, Helv. Chim. Acta 5, 147 (1922)] in boiling C<sub>6</sub>H<sub>6</sub> in the absence of air gave I, violet-black crystals, m. 240-51° (from C<sub>6</sub>H<sub>6</sub>, under an inert atmospheric), not paramagnetic and existing as the o-quinoid rather than

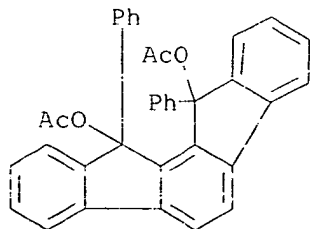
the diradical form. Crystalline I is stable in air, but only without air in solution, is not affected by radiation or light in Et<sub>2</sub>O or C<sub>6</sub>H<sub>6</sub>, is brominated when cold to IV, and forms with maleic anhydride the 11,12-adduct, colorless prisms, m. 315°, reduced to isomers of I by Zn in HOAc while KOH in EtOH gives 1 isomer below the m.p. From previous work (C.A. 49, 12418i) benz[c]indeno[2,1-a]fluorene-13,14-dione (VII) treated with PhMgBr gave the 2-stereoisomeric 13,14-diphenyl-13,14-dihydroxybenz[c]inden[2,1-a]fluorenes (VIII), colorless rods, m. 300°, and colorless prisms, m. 348°, with some mono-Ph by-product, red rods, m. 290°. VIII with HBr in HOAc gave 13,14-dibromo-13,14-diphenylbenz[c]indeno [2,1-a]fluorene (IX), yellow prisms, m. 254-6°. Hydrolysis of IX gave VII above the fusion temperature and reduction with Zn in HOAc yielded the 2 stereoisomeric 13,14-dihydro-13,14-diphenylbenz [c]indeno [2,1-a] fluorenes (X), colorless needles m. 294°, and colorless rods, m. 282°, separable by chromatography, also obtainable from KI in HOAc and VIII. II can be obtained as a green solution in the absence of air from IX with Cu in C<sub>6</sub>H<sub>6</sub>; it gives the maleic anhydride adduct, m. 370°, and is considered to be very unstable because of the large number of conjugated

double bonds present in the mol.

CC 10 (Organic Chemistry)  
 IT 119925-92-7P, Benz[c]indeno[2,1-a]fluoren-13(14H)-one,  
 14-hydroxy-14-phenyl- 120267-44-9P, Indeno[2,1-a]fluorene,  
 11,12-diphenyl- **120267-45-0P**, Indeno[2,1-a]fluorene,  
 11,12-dibromo-11,12-dihydro-11,12-diphenyl- 121815-01-8P,  
 Benz[c]indeno[2,1-a]fluorene, 13,14-dihydro-13,14-diphenyl-, stereoisomers  
**121967-72-4P**, Indeno[2,1-a]fluorene-11,12-diol,  
 11,12-dihydro-11,12-diphenyl-, diacetate 122273-24-9P,  
 Benz[a]indeno[1,2,3-fg]aceanthrylene-9,10-dicarboxylic anhydride,  
 8b,9,10,10a-tetrahydro-8b,10a-diphenyl- 124289-38-9P,  
 Benz[c]indeno[2,1-a]fluorene-13,14-diol, 13,14-dihydro-13,14-diphenyl-,  
 stereoisomers 124290-45-5P, Indeno[1,2,3-cd]fluoranthene-5,6-  
 dicarboxylic anhydride, 4b,5,6,6a-tetrahydro-4b,6a-diphenyl-  
 124513-91-3P, Benz[c]indeno[2,1-a]fluorene, 13,14-dibromo-13,14-dihydro-  
 13,14-diphenyl- 125614-11-1P, Benz[c]indeno[2,1-a]fluorene,  
 13,14-diphenyl- **876504-70-0P**, Indeno[2,1-a]fluoren-11(12H)-one,  
 12-hydroxy-12-phenyl-  
 RL: PREP (Preparation)  
 (preparation of)  
 IT **124180-22-9**, Indeno[2,1-a]fluorene-11,12-diol,  
 11,12-dihydro-11,12-diphenyl- **124180-97-8**, Indeno[2,1-  
 a]fluorene, 11,12-dihydro-11,12-diphenyl-  
 (stereoisomers)  
 IT **120267-45-0P**, Indeno[2,1-a]fluorene, 11,12-dibromo-11,12-dihydro-  
 11,12-diphenyl- **121967-72-4P**, Indeno[2,1-a]fluorene-11,12-diol,  
 11,12-dihydro-11,12-diphenyl-, diacetate **876504-70-0P**,  
 Indeno[2,1-a]fluoren-11(12H)-one, 12-hydroxy-12-phenyl-  
 RL: PREP (Preparation)  
 (preparation of)  
 RN 120267-45-0 HCAPLUS  
 CN Indeno[2,1-a]fluorene, 11,12-dibromo-11,12-dihydro-11,12-diphenyl- (CA  
 INDEX NAME)



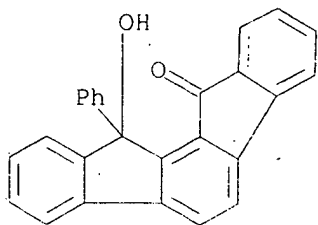
RN 121967-72-4 HCAPLUS  
 CN Indeno[2,1-a]fluorene-11,12-diol, 11,12-dihydro-11,12-diphenyl-, diacetate  
 (6CI) (CA INDEX NAME)





RN 876504-70-0 HCAPLUS

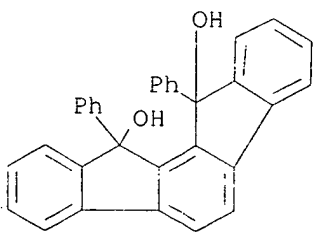
CN Indeno[2,1-a]fluorene-11(12H)-one, 12-hydroxy-12-phenyl- (CA INDEX NAME)



IT **124180-22-9**, Indeno[2,1-a]fluorene-11,12-diol, 11,12-dihydro-11,12-diphenyl- **124180-97-8**, Indeno[2,1-a]fluorene, 11,12-dihydro-11,12-diphenyl- (stereoisomers)

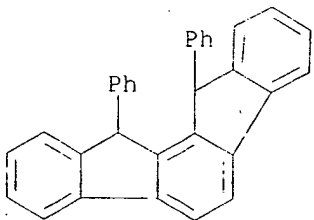
RN 124180-22-9 HCAPLUS

CN Indeno[2,1-a]fluorene-11,12-diol, 11,12-dihydro-11,12-diphenyl- (CA INDEX NAME)



RN 124180-97-8 HCAPLUS

CN Indeno[2,1-a]fluorene, 11,12-dihydro-11,12-diphenyl- (CA INDEX NAME)



L96 ANSWER 16 OF 20 HCAPLUS COPYRIGHT 2007 ACS on STN

AN 1957:1708 HCAPLUS

DN 51:1708

OREF 51:345b-d

TI Dihydroperoxide derivatives of indenofluorene

AU Le Berre, Andre

SO Compt. rend. (1956), 242, 2576-9

DT Journal

LA Unavailable

AB Treatment of 11,12-diphenyl-11,12-dibromo-11,12-dihydroindeno[2,1- $\alpha$ ]fluorene (I) with 80% Superoxol in dioxane in the presence of Na<sub>2</sub>CO<sub>3</sub> yields 2 isomers of 11,12-diphenyl-11,12-dihydroperoxy-11,12-

dihydroindeno[2,1- $\alpha$ ]fluorene (II). The benzo[c] derivative (III) of I yields but one isomer of 13,14-diphenyl-13,14-dihydroperoxy-13,14-dihydrobenzo[c]indeno[2,1- $\alpha$ ]fluorene (IV). The major isomer of II seps. with 0.5 mole and the other with 2 moles C<sub>6</sub>H<sub>6</sub>. The desolvated materials m. 260-2° (decomposition) and 234-6° (decomposition), resp. IV seps. with 0.5 mole ether, m. 180°, or 2 moles Me<sub>2</sub>CO, m. 170°; it undergoes decomposition on desolvation. In the presence of Pb(OAc)<sub>4</sub>, II is converted to 1-benzoyl-2-(o-hydroxyphenyl)-9-phenyl-9-hydroxyfluorene (V). The mechanism is postulated. IV is converted to the corresponding hydroxyfluorene. II is stable in AcOH, but a trace of HCl or H<sub>2</sub>SO<sub>4</sub> converts it to V. In similar circumstances IV resinifies completely. Pyrolysis of II and IV yield, by loss of the peroxide O, the corresponding diols in 35% yield; these m. 276° (decomposition) and 348° (decomposition), resp.

CC 10 (Organic Chemistry)

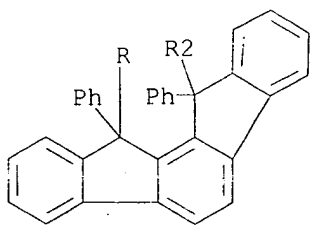
IT **124145-09-1**, Hydroperoxide, (11,12-dihydro-11,12-diphenylindeno[2,1- $\alpha$ ]fluoren-11,12-ylene)di- (isomers)

IT 103267-02-3P, Ketone, 9-hydroxy-2-(o-hydroxyphenyl)-9-phenylfluoren-1-yl phenyl **124180-22-9P**, Indeno[2,1- $\alpha$ ]fluorene-11,12-diol, 11,12-dihydro-11,12-diphenyl- 124269-74-5P, Hydroperoxide, (13,14-dihydro-13,14-diphenylbenz[c]indeno[2,1- $\alpha$ ]fluoren-13,14-ylene)di- 124289-38-9P, Benz[c]indeno[2,1- $\alpha$ ]fluorene-13,14-diol, 13,14-dihydro-13,14-diphenyl- 124514-15-4P, Ketone, 7-hydroxy-5-(o-hydroxyphenyl)-7-phenyl-7H-benzo[c]fluoren-6-yl phenyl  
RL: PREP (Preparation)  
(preparation of)

IT **124145-09-1**, Hydroperoxide, (11,12-dihydro-11,12-diphenylindeno[2,1- $\alpha$ ]fluoren-11,12-ylene)di- (isomers)

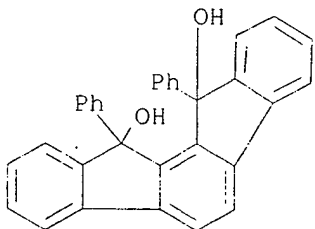
RN 124145-09-1 HCAPLUS

CN Hydroperoxide, (11,12-dihydro-11,12-diphenylindeno[2,1- $\alpha$ ]fluoren-11,12-ylene)di- (6CI) (CA INDEX NAME)



IT **124180-22-9P**, Indeno[2,1- $\alpha$ ]fluorene-11,12-diol, 11,12-dihydro-11,12-diphenyl-  
RL: PREP (Preparation)

(preparation of)  
 RN 124180-22-9 HCAPLUS  
 CN Indeno[2,1-a]fluorene-11,12-diol, 11,12-dihydro-11,12-diphenyl- (CA INDEX NAME)



L96 ANSWER 17 OF 20 HCAPLUS COPYRIGHT 2007 ACS on STN

AN 1956:73861 HCAPLUS

DN 50:73861

OREF 50:13857d-g

TI Orthoquinoidal indenofluorene hydrocarbons. Their autoxidation in neutral mediums

AU Etienne, Andre; Le Berre, Andre

SO Compt. rend. (1956), 242, 1899-1901

DT Journal

LA Unavailable

AB The behavior of 11,12-diphenylindeno[2,1-a]fluorene (I) and 13,14-diphenylbenz[c]indeno[2,1-a]fluorene (II) toward O has been studied. A dark violet solution of I in C<sub>6</sub>H<sub>6</sub>, CS<sub>2</sub>, or Et<sub>2</sub>O reacted with O in the absence of light to give an orange brown solution which on evaporation to dryness

gave a yellow resin, very soluble in most organic solvents except alc. and cyclohexane. The resin did not give O on heating although its percentage composition corresponded to that of a cyclic endoperoxide. It liberated iodine from HOAc-KI solution and was postulated to be a complex mixture of **polymerized** peroxides. This autoxidation was not facilitated by light and proceeded only until 1 mole O had been absorbed. The intermediate cyclic peroxide could not be isolated. The autoxidation of I in the presence of antioxidants was also investigated. With o,o'-di-tert-butyl-p-cresol, the same yellow resin was obtained, and with excess hydroquinone or pyrogallol, autoxidation occurred, followed by H addition to yield 11,12-diphenyl-11,12-dihydroxy-11,12-dihydroindeno[2,1-a]fluorene, m. 276° (10% with hydroquinone, 70% with pyrogallol). II behaved similarly to I, its dark green solution being decolorized instantly on exposure to air to give a resin from which 10-15% 13,14-diphenyl-13,14-dihydroxy-13,14-dihydrobenz[c]-indeno[2,1-a]fluorene, m. 348°, was isolated. II presumably also formed an intermediate unstable cyclic peroxide which was more reactive than that from I since no H donor was necessary to form the diol. The autoxidation of I and II differs from that of the acenes in that the acenes require light activation and form peroxides stable enough to be isolated.

CC 10 (Organic Chemistry)

IT 124180-22-9P, Indeno[2,1-a]fluorene-11,12-diol, 11,12-dihydro-11,12-diphenyl- 124289-38-9P, Benz[c]indeno[2,1-a]fluorene-13,14-diol, 13,14-dihydro-13,14-diphenyl-

RL: PREP (Preparation)

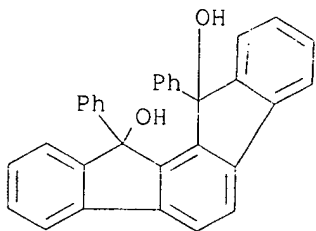
(preparation of)

IT 124180-22-9P, Indeno[2,1-a]fluorene-11,12-diol, 11,12-dihydro-11,12-diphenyl-

RL: PREP (Preparation)  
(preparation of)

RN 124180-22-9 HCAPLUS

CN Indeno[2,1-a]fluorene-11,12-diol, 11,12-dihydro-11,12-diphenyl- (CA INDEX NAME)



L96 ANSWER 18 OF 20 HCAPLUS COPYRIGHT 2007 ACS on STN

AN 1952:42288 HCAPLUS

DN 46:42288

OREF 46:7085f-i,7086a-c

TI Fluorenacenes and fluorenaphenes. Syntheses in the indenofluorene series.  
II. endo-cis-Fluorenaphene (indeno[2,1-a]fluorene) and trans-fluorenacene  
(indeno[1,2-b]-fluorene)

AU Deuschel, Werner

CS Univ. Fribourg, Fribourg, Switz.

SO Helvetica Chimica Acta (1951), 34, 2403-16

CODEN: HCACAV; ISSN: 0018-019X

DT Journal

LA German

OS CASREACT 46:42288

GI For diagram(s), see printed CA Issue.

AB cf. C.A. 45, 10224e. A mixture of  $\text{AlCl}_3$  and  $\text{CS}_2$  are refluxed and the condensed liquid returned through 3,6,1,2- $\text{Ph}_2\text{C}_6\text{H}_2(\text{CO})_2\text{O}$ , forming 9-oxo-2-phenyl-1-fluorene-11-carboxylic acid (I), m. 198-200°. I,  $\text{SOCl}_2$ , and a little concentrated  $\text{H}_2\text{SO}_4$  in refluxing  $\text{CCl}_4$  form an orange-yellow precipitate of indeno[2,1-a]fluorene-11,12-dione, m. 300° after sublimation at  $5 + 10^{-3}$  mm., and 220° (nos. after other m.ps. will be the pressure and temperature of purifying sublimation), which after distillation from Zn at 10 mm. and 360° forms indeno[2,1-a]fluorene (II), m. 286.5-7.5° ( $5 + 10^{-3}$  180°). Cyclohexene, p- $\text{C}_6\text{H}_4\text{Me}_2$ ,  $\text{CS}_2$ , and  $\text{AlCl}_3$  form 2,5,1,4-( $\text{C}_6\text{H}_{11}$ ) $2\text{C}_6\text{H}_2\text{Me}_2$  (III), m. 154-5° (10, 150°), which, heated with Se at 340-50° or with 10% Pd-C at 280-90° forms 2,5,1,4- $\text{Ph}_2\text{C}_6\text{H}_2\text{Me}_2$  (IV), m. 182-4° (10, 170°). Chlorination of IV at 185° in an apparatus (diagram given) in which the Cl is produced from  $\text{KMnO}_4$  and HCl and the HCl produced recycled to avoid waste of Cl forms 6,6,12,12-tetrachloroindeno[1,2-b]fluorene (V), decompose 330°. V refluxed with  $\text{PhNO}_2$ ,  $\text{H}_2\text{O}$ , and a little  $\text{CuCl}_2$  forms violet-red indeno[1,2-b]fluorene-6,12-dione (VI), m. 345-6° ( $5 + 10^{-3}$ , 230°); bis(phenylhydrazone), decompose 273-5°. Heating VI, Zn, HOAc, and  $\text{C}_5\text{H}_5\text{N}$  forms indeno[1,2-b]fluorene (VII), m. 298-300°, isolated by pouring into 2 N HCl and filtration. Dehydrogenation of IV in a special apparatus (construction details given) in which the vapors of the high-boiling material pass (at 120 mm. and 400°) over 20% Pd-C and are returned within the apparatus itself to be recycled forms VII, m. 300-2° ( $5 + 10^{-3}$ , 180-90°), identical (mixed m.p.) with the preparation above and giving V on chlorination. An attempt was made to prepare VII via condensation of 2-(phenylethynyl)indene (VIII) with maleic acid to the

compound (IX) or with EtO<sub>2</sub>CCH<sub>2</sub>CHO to the compound (X), both of which should give VII by reactions similar to those used in the preparation of II. VIII, m. 98-100° (5 + 10<sup>-3</sup>, 95°), was obtained from 2-indanone and PhC.tplbond.CMgBr or PhC.tplbond.CLi and dehydration of the intermediate alc., m. 86.5-7.5° (5 + 10<sup>-3</sup>, 80°), with p-MeC<sub>6</sub>H<sub>4</sub>SO<sub>3</sub>H. But the condensations either did not take place or went to **polymeric** glassy masses. Attempts to prepare VIII from PhC.tplbond.CNa and 2-bromo-1-indanol were not successful. Oxidation of III is very difficult. CrO<sub>3</sub> in Ac<sub>2</sub>O, followed by KMnO<sub>4</sub>, gave a small amount of acid, decompose 250-60°, which with warm concentrated H<sub>2</sub>SO<sub>4</sub> gave a violet precipitate, VI(?). The acid was desired to check the synthesis by decarboxylating to p-C<sub>6</sub>H<sub>4</sub>Ph<sub>2</sub>.

CC 10 (Organic Chemistry)

IT 486-52-2P, Indeno[1,2-b]fluorene, 6,12-dihydro- **3074-14-4P**,  
Indeno[2,1-a]fluorene-11,12-dione 4516-08-9P, p-Xylene,  
2,5-dicyclohexyl- 5695-13-6P, Indeno[1,2-b]fluorene-6,12-dione  
**5815-59-8P**, Indeno[2,1-a]fluorene, 11,12-dihydro- 20260-22-4P,  
p-Terphenyl, 2',5'-dimethyl- 102184-79-2P, 1-Fluorene-carboxylic acid,  
9-oxo-2-phenyl- 119682-10-9P, Indeno[1,2-b]fluorene-6,12-dione,  
bis(phenylhydrazone) 408313-70-2P, Indeno[1,2-b]fluorene,  
6,6,12,12-tetrachloro-6,12-dihydro-

RL: PREP (Preparation)

(preparation of)

IT **3074-14-4P**, Indeno[2,1-a]fluorene-11,12-dione **5815-59-8P**

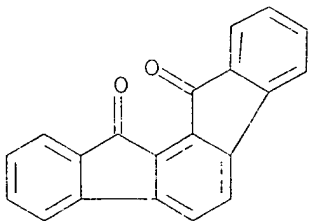
, Indeno[2,1-a]fluorene, 11,12-dihydro-

RL: PREP (Preparation)

(preparation of)

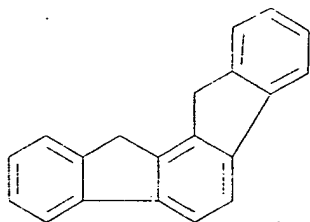
RN 3074-14-4 HCAPLUS

CN Indeno[2,1-a]fluorene-11,12-dione (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)



RN 5815-59-8 HCAPLUS

CN Indeno[2,1-a]fluorene, 11,12-dihydro- (CA INDEX NAME)



L96 ANSWER 19 OF 20 HCAPLUS COPYRIGHT 2007 ACS on STN

AN 1939:29837 HCAPLUS

DN 33:29837

OREF 33:4232c-i,4233a-c

TI Phenylated phthalic acids and anthracene derivatives

AU Weizmann, Ch.; Bergmann, Ernst; Haskelberg, L.

SO Journal of the Chemical Society (1939) 391-7

CODEN: JCSOA9; ISSN: 0368-1769

DT Journal

LA Unavailable

OS CASREACT 33:29837

AB 3,6-Diphenyltetrahydrophthalic anhydride and S, heated at 260-70° for 20 min., give a nearly quant. yield of 3,6-diphenylphthalic anhydride (I), m. 224°; solution in Na<sub>2</sub>CO<sub>3</sub> and acidification give the free acid, m. 162° (decomposition), whose Me ester m. 188°. I and PhMgBr in xylene give 50% of 2-benzoyl-3,6-di-phenylbenzoic acid (II), m. 167° (concentrated H<sub>2</sub>SO<sub>4</sub> gives a dark green color); α-C<sub>10</sub>H<sub>7</sub>MgBr gives 50% of the 2-α-naphthoyl homolog of II, m. 188°; p-MeOC<sub>6</sub>H<sub>4</sub>MgBr gives the 2-p-methoxybenzoyl homolog, with 2 mol of H<sub>2</sub>O, decomps. 125° and then m. 175° (Me ester, m. 185°), gives with concentrated H<sub>2</sub>SO<sub>4</sub> a brown-violet color; 2-(6'-methoxy-β-naphthoyl) homolog, m. 220° (Me ester, 220°), gives with H<sub>2</sub>SO<sub>4</sub> a dark green color; 2-p-bromobenzoyl homolog, m. 200° (intense green color with H<sub>2</sub>SO<sub>4</sub>). Cyclization of the keto acids proved very tedious; the presence of the voluminous o-substituents slows down the ring closure so that side reactions may prevail; e. g., sulfonation readily takes place with H<sub>2</sub>SO<sub>4</sub> as the cyclizing agent. I (10 g.) and 5 g. CO(NH<sub>2</sub>)<sub>2</sub>, heated 2 h. at 200°, give 8.2 g. of 3,6-diphenylphthalimide (III), m. 245°; this could not be degraded to an NH<sub>2</sub> acid by NaOCl or NaOBr. I and NH<sub>2</sub>OH in absolute EtOH, allowed to stand 30 h. at room temperature, give a quant. yield of the N-HO derivative of III,

m.

238°; heating with dilute NaOH for 12 h. at 100° gives 96% of 3,6-diphenylantranilic acid, m. 200° (decomposition); Me ester, m. 119-20°; Ac derivative, m. 215°; the acid does not react with ClCH<sub>2</sub>CO<sub>2</sub>H, BrCH<sub>2</sub>CO<sub>2</sub>H, HCHO or KCN and therefore the synthesis of tetraphenylindigotin could not be effected. I (6 g.) and AlCl<sub>3</sub> in C<sub>6</sub>H<sub>6</sub>, boiled 2 h., give 4.5 g. of 2-Phenylfluorenone-1-carboxylic acid (IV), m. 199-201° (concentrated H<sub>2</sub>SO<sub>4</sub> gives a red color); phenylhydrazone, yellow, m. 177°; Me ester (V), yellow, m. 142°. V (3.1 g.) and PhMgBr give 1 g. of 1-benzoyl-2-phenylfluorenone (VI), m. 236° (concentrated H<sub>2</sub>SO<sub>4</sub> gives a violet-red color). V (3.1 g.) and PhLi after 24 h. give 2 g. of 1-hydroxybenzohydryl-2,9-diphenylfluorenone, m. 123° (decomposition), and 1 g. of VI. Decarboxylation of IV with Cu bronze in boiling quinoline gives 2-phenylfluorenone, golden, m. 140-1° (H<sub>2</sub>SO<sub>4</sub> gives a deep red solution); phenylhydrazone, brown, m. 168°. Boiling IV with SOCl<sub>2</sub> in CCl<sub>4</sub> for 4 h. gives 1'-ketoindeno(2',3',1,2)fluorenone, golden-red, m. 298°; bisphenylhydrazone, brown, m. 215° (decomposition). 3-PhC<sub>6</sub>H<sub>3</sub>(CO)<sub>2</sub>O (prepared from the tetrahydro derivative and S at 280° for 3 h.) and PhMgBr give a mixture of 2-benzoyl-3-phenylbenzoic acid, m. 172°, and the 6-benzoyl-2-Ph isomer, m. 163°. 1-Phenylantraquinone (VII) and PhMgBr give 9,10-dihydroxy-1,9,10-triphenyl-9,10-dihydroanthracene, m. 238° (dark blue color reaction with concentrated H<sub>2</sub>SO<sub>4</sub>). Reduction of VII with Zn in NaOH-NH<sub>4</sub>OH gives 1-phenylantracene, yellow, m. 123°. 1-Phenylbutadiene and C<sub>6</sub>H<sub>4</sub>O<sub>2</sub>, boiled in xylene for 6 h., give a mixture, separated by AcOH, of 1,5-diphenyl-1,4,5,8,11,12,13,14-octahydroanthraquinone (VIII), m. 230°, and 5-phenyl-5,8,9,10-tetrahydro-α-naphthaquinone, m. 170° (blood-red color with concentrated H<sub>2</sub>SO<sub>4</sub>). Passing air through VIII in 15% EtOH-KOH gives 1,5-diphenylantraquinone, m. 355°. 1,4-Diphenylbutadiene (IX) and C<sub>6</sub>H<sub>4</sub>O<sub>2</sub>, heated under slightly reduced pressure until a violent reaction sets in and kept at that temperature for 5 h., give 1,4,5,8-tetraphenylantraquinone, m. 355°; PhLi in Et<sub>2</sub>O (24 h.) gives 9,10-dihydroxy-1,4,5,8,9,10-

hexaphenyl-9,10-dihydroanthracene, m. above 370°, very slightly soluble IX and  $\alpha$ -naphthaquinone, heated 10 h. at 160°, give 1,4-diphenylanthraquinone (X), yellow, m. 212°; di-NO<sub>2</sub> compound, m. 208°; H<sub>2</sub>SO<sub>4</sub> at 100° gives a disulfonic acid, isolated as the Na salt (with 3 mol of H<sub>2</sub>O); Br at room temperature gives a di-Br derivative, yellow, m. 295°, whereas at 60° there is formed a tetra-Br derivative, red, m. above 300°. Reduction of X with Zn in NaOHNH<sub>4</sub>OH gives a mixture of 1,4-diphenylanthracene, yellow, m. 170° (picrate, brown, m. 173°), and 9-hydroxy-1,4-diphenyl-9,10-dihydroanthracene, m. 155°. X and PhMgBr give 9,10-dihydroxy-1,4,9,10-tetraphenyl-9,10-dihydroanthracene, m. 240°; dehydration with HCO<sub>2</sub>H or AcCl gives a hydrocarbon (XI), C<sub>38</sub>H<sub>26</sub>, m. 322°; reduction with KI and Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub> gives a mixture of XI and 1,4,9,10-tetraphenylanthracene, yellow, m. 205°; boiling with HCO<sub>2</sub>H gives XI.

CC 10 (Organic Chemistry)

IT 1162-64-7P, p-Terphenyl-2',3'-dicarboxylic anhydride 1162-64-7P, Phthalic anhydride, 3,6-diphenyl- 1169-58-0P, p-Terphenyl-2',3'-dicarboxylic acid, dimethyl ester 1169-58-0P, Phthalic acid, 3,6-diphenyl-, dimethyl ester 1714-09-6P, Anthracene, 1-phenyl- 1714-16-5P, Anthracene, 1,4-diphenyl- 1714-23-4P, Anthracene, 1,4,9,10-tetraphenyl- 3074-14-4P, Indeno[2,1-a]fluorene-11,12-dione 3096-49-9P, 9-Fluorenone, 2-phenyl- 19799-46-3P, p-Terphenyl-2',3'-dicarboxylic acid 19799-46-3P, Phthalic acid, 3,6-diphenyl- 24024-98-4P, 2-Biphenylcarboxylic acid, 3-benzoyl- 75312-13-9P, Anthraquinone, 1,5-diphenyl- 97692-17-6P, Anthraquinone, 1,4,5,8-tetraphenyl- 108842-67-7P, 1,4-Naphthoquinone, 4a,5,8,8a-tetrahydro-5-phenyl- 412025-97-9P, p-Terphenyl-2'-carboxylic acid, 3'-benzoyl- 412297-69-9P, p-Terphenyl-2',3'-dicarboximide, N-hydroxy- 412297-69-9P, Phthalimide, N-hydroxy-3,6-diphenyl- 419567-61-6P, Anthraquinone, 1,4,4a,5,8,8a,9a,10a-octahydro-1,5-diphenyl- 854396-36-4P, Dibenzo[c,lm]fluorene, 8,12b-dihydro-7,8,12b-triphenyl- 855254-49-8P, 3-Biphenylcarboxylic acid, 2-benzoyl- 857588-46-6P, 9-Anthrol, 9,10-dihydro-1,4-diphenyl- 857589-44-7P, Anthranilic acid, N-acetyl-3,6-diphenyl- 857589-44-7P, p-Terphenyl-2'-carboxylic acid, 3'-acetamido- 857589-54-9P, Anthracene, 9,10-dihydro-9,10-dimethoxy-1,4,9,10-tetraphenyl- 857589-97-0P, 9,10-Anthradiol, 9,10-dihydro-1,9,10-triphenyl- 858799-55-0P, 9-Fluorenone, 2-phenyl-, phenylhydrazine 858799-77-6P, 9-Fluorenone, 1-benzoyl-2-phenyl- 858800-06-3P, 1-Fluorenenemethanol, 9-hydroxy- $\alpha,\alpha,2,9$ -tetraphenyl- 860242-54-2P, p-Terphenyl-2'-carboxylic acid, 3'-anisoyl-, Me ester 860242-63-3P, p-Terphenyl-2'-carboxylic acid, 3'-anisoyl- 860242-71-3P, p-Terphenyl-2'-carboxylic acid, 3'-amino- 860242-71-3P, Anthranilic acid, 3,6-diphenyl- 860248-80-2P, p-Terphenyl-2'-carboxylic acid, 3'-(1-naphthoyl)- 860248-88-0P, p-Terphenyl-2'-carboxylic acid, 3'-(6-methoxy-2-naphthoyl)-, Me ester 860248-95-9P, p-Terphenyl-2'-carboxylic acid, 3'-(6-methoxy-2-naphthoyl)- 860249-20-3P, p-Terphenyl-2'-carboxylic acid, 3'-(p-bromobenzoyl)- 860530-81-0P, Anthranilic acid, 3,6-diphenyl-, Me ester 860530-85-4P, 9,10-Anthradiol, 9,10-dihydro-1,4,9,10-tetraphenyl- 860530-88-7P, 9,10-Anthradiol, 9,10-dihydro-1,4,5,8,9,10-hexaphenyl- 860531-64-2P, Anthracene, 1,4-diphenyl-, picrate 861018-66-8P, Phthalimide, 3,6-diphenyl-

RL: PREP (Preparation)

(preparation of)

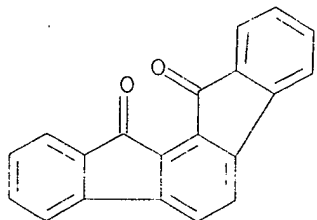
IT 3074-14-4P, Indeno[2,1-a]fluorene-11,12-dione

RL: PREP (Preparation)

(preparation of)

RN 3074-14-4 HCAPLUS

CN Indeno[2,1-a]fluorene-11,12-dione (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)



L96 ANSWER 20 OF 20 HCAPLUS COPYRIGHT 2007 ACS on STN

AN 1935:13987 HCAPLUS

DN 29:13987

OREF 29:17991,1800a-f

TI Highly arylated aromatic compounds. III. Highly phenylated benzenecarboxylic acids and their transformation products

AU Dilthey, W.; Thewalt, I.; Trosken, O.

SO Berichte der Deutschen Chemischen Gesellschaft [Abteilung] B: Abhandlungen (1934), 67B, 1959-64

CODEN: BDCBAD; ISSN: 0365-9488

DT Journal

LA Unavailable

AB cf. C. A. 28, 3733.7; Allen and Sheps, C. A. 29, 142.1.

endo-Carbonyltetraphenyldihydrophthalic anhydride (I), m. 223° (C. A. 28, 4728), is obtained in 12 g. yield from 12 g. finely powdered tetraphenylcyclopentadienone (called tetracyclone for short) slowly added to 3 g. maleic anhydride in 100 cc. benzene and boiled 7 hrs. If PhCl (b. 132°) is used as the solvent instead of benzene, there is obtained 10-11 g. of tetraphenyldibydrophthalic anhydride (II), m. 243°, also obtained from I boiled in PhCl. In PhNO<sub>2</sub>, instead of benzene or PhCl the product is tetraphenylphthalic anhydride (III) (9 g.), m. 295° (occasionally 286°) after driving off the benzene of crystallization; the phthalic acid (IV), from III with boiling alc. KOH, m. 286°. III is conveniently prepared (35 g. yield) by slowly stirring 48 g. II into 150 g. molten S at 250° and slowly raising the temperature to 300° and keeping it at that point 2 hrs. I, II and III can also be prepared without solvents but the temps. must be carefully controlled. The accumulation of Ph groups in IV markedly decreases the reactivity of the CO<sub>2</sub>H groups. IV still forms salts but it is practically no longer soluble in aqueous NH<sub>4</sub>OH and yields no Et ester by the ordinary methods. Its esters are readily obtained, however, by addition of the corresponding acetylenedicarboxylates to tetracyclone. Thus, the di-Me ester, m. 258°, is obtained in 5.2 g. yield from 5 g. tetracyclone and 2.5 g. (.tplbond. CCO<sub>2</sub>Me)<sub>2</sub> cautiously heated to 160° (this ester is also obtained quant. from IV and CH<sub>2</sub>N<sub>2</sub> in MeOH-Et<sub>2</sub>O); di-Et ester (10 g. from 3.4 g. (.tplbond. CCO<sub>2</sub>Et)<sub>2</sub>), m. 205-6° (A. and S. give 187-8°), cannot be saponified III (1 g.), heated 10 min. in benzene with 0.8 g. AlCl<sub>3</sub>, gives 0.7 g. triphenylfluorenonecarboxylic acid, lemon-yellow, m. 322°, converted back into IV by fusion with alkali; K salt, lemon-yellow, m. around 370°; oxime, pale yellow, m. 300°. Boiled 2 hrs. in benzene with 3 g. AlCl<sub>3</sub>, 2 g. III yields 1.8 g. diphenyldifluorenone, orange, m. 396°, also obtained from III and fused ZnCl<sub>2</sub> if too high heating is avoided, from III allowed to stand in concentrated H<sub>2</sub>SO<sub>4</sub>, and from III with PCl<sub>5</sub>; dioxime, deep orange-red, carbonizes before it melts. Me pentaphenylbenzoate (6.2 g. from 2 g. PhC .tplbond. CCO<sub>2</sub>Me and tetracyclone at 170-5°, or from Ph<sub>5</sub>C<sub>6</sub>CO<sub>2</sub>H and CH<sub>2</sub>N<sub>2</sub> in C<sub>6</sub>H<sub>6</sub>MeOH), m. 342°, cannot be saponified Tetraphenylfluorenone (3 g. from 4 g. Ph<sub>5</sub>C<sub>6</sub>CO<sub>2</sub>H with POCl<sub>3</sub>-PCl<sub>5</sub> on the water bath), yellow, m. 298°, shows brownish yellow halochromism in hot concentrated H<sub>2</sub>SO<sub>4</sub>. Et



tetraphenyl-o-toluate (2.2 g. from 1.5g. MeC.tplbond.CCO2Et and tetracyclone at 200°), m. 205°; free acid, similarly obtained from MeC.tplbond.CCO2H, m. 302°.

CC 10 (Organic Chemistry)

IT 1487-12-3P, Benzene, 1,2,3,4-tetraphenyl- 2857-85-4P, Benzoic acid, pentaphenyl-, methyl ester 3383-32-2P, Benzene, 1,2,4,5-tetraphenyl- 4741-53-1P, Phthalic anhydride, tetraphenyl- 6583-73-9P, 9-Fluorenone, 1,2,3,4-tetraphenyl- 6971-41-1P,  $\Delta^3,5-1,2$ -Cyclohexadienedicarboxylic anhydride, 3,4,5,6-tetraphenyl- **38023-71-1P**, Benzo[1,2-a,4,3-a']diindene-11,12-dione, 5,6-diphenyl- 202828-34-0P, Bicyclo[2.2.1]hept-5-ene-2,3-dicarboxylic anhydride, 7-keto-1,4,5,6-tetraphenyl- 628727-72-0P, o-Toluic acid, 3,4,5,6-tetraphenyl-, ethyl ester 858842-80-5P, o-Toluic acid, 3,4,5,6-tetraphenyl- **860598-76-1P**, Benzo[1,2-a,4,3-a']diindene-11,12-dione, 5,6-diphenyl-, dioxime  
RL: PREP (Preparation)

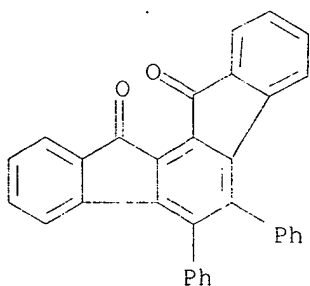
(preparation of)

IT **38023-71-1P**, Benzo[1,2-a,4,3-a']diindene-11,12-dione, 5,6-diphenyl- **860598-76-1P**, Benzo[1,2-a,4,3-a']diindene-11,12-dione, 5,6-diphenyl-, dioxime  
RL: PREP (Preparation)

(preparation of)

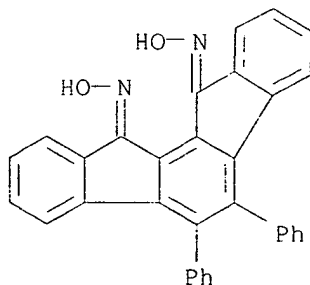
RN 38023-71-1 HCAPLUS

CN Indeno[2,1-a]fluorene-11,12-dione, 5,6-diphenyl- (CA INDEX NAME)



RN 860598-76-1 HCAPLUS

CN Benzo[1,2-a,4,3-a']diindene-11,12-dione, 5,6-diphenyl-, dioxime (3CI) (CA INDEX NAME)



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## IN THE CLAIMS

Please amend the claims as follows:

1. (Currently amended) A method for manufacturing the imide-substituted polymer polymers comprising the steps of:

copolymerization step done by dividing the feed into the Mixture (A) composed of aromatic vinyl monomers, initiators, and chain transfer agents and the Mixture (B) composed of unsaturated dicarboxylic anhydride monomers and solvents, and then charging copolymerization reactors simultaneously with them while adjusting the flow rate of each mixture according to the compositional ratio of the feed, and finally copolymerizing aromatic vinyl monomers and unsaturated dicarboxylic anhydride monomers in the copolymerization reactors, wherein the mole ratio of the aromatic vinyl monomers to the unsaturated dicarboxylic anhydride monomers is 0.67-6.0:1;

separation step performed by supplying the polymerized solution discharged from the copolymerization reactors into a separator continuously, and then removing unreacted monomers and solvents sufficiently;

imide substitution step accomplished by supplying the polymer melt discharged from the separator continuously into imide substitution reactors and adding continuously the Mixture (C) composed of primary amines, catalysts for an imide substitution reaction, and solvents at the same time, and then reacting unsaturated dicarboxylic anhydride units in said copolymers with the primary amines; and

devolatilization step done by removing low-molecular-weight volatiles from the polymer solution discharged from the imide substitution reactors in ~~the~~ a devolatilizer, wherein the imide-substituted polymer includes 40-55 wt% of aromatic vinyl units, 0-5 wt% of unsaturated dicarboxylic anhydride units, and 40-60 wt% of the imidized units from unsaturated dicarboxylic anhydride units and the residence time in the copolymerization step is within the range of 2.0 to 5.0 hours.

2. (Original) The method for manufacturing the imide-substituted polymer according to claim 1, wherein said aromatic vinyl monomer in the Mixture (A) is selected